A STUDY ON TAPERED OPTICAL FIBER SENSOR FOR RELATIVE HUMIDITY MEASUREMENTS

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FACULTY OF ENGINEERING
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A STUDY ON TAPERED OPTICAL FIBER SENSOR FOR RELATIVE HUMIDITY MEASURMENTS

ABSTRACT

In this project, the effects of coating material which are PVA and PMMA on the tapered fiber towards relative humidity sensitivity were studied. Flame brushing was used to shape the fiber into tapered fiber. The diameters of the samples used in the experiment were in the range between 2 - 10 μm. The power output of each sample was measured while the proposed sensor is in a locked chamber when relative humidity increased between 35-85% RH. The sensors with diameter below 4 μm showed good sensitivity with fine resolution. The sample of diameter 2.6 μm had the highest sensitivity, finest resolution and smallest standard deviation. Thus, the sample with diameter 2.6 μm was selected for the coating process. The PVA coated sample had the best sensitivity, linearity and resolution followed by the bare fiber and the PMMA coated sample. Next, the stability of the three samples, the bare, PVA coated and PMMA coated was tested. The power output of each sample was continuously measured in constant relative humidity inside the locked chamber. All the chambers showed good and stable performance. However, the PVA coated sample was the most consistent. Finally, the response and recovery time of the three samples was measured. Each sample was moved between two chambers with different relative humidity and the time taken for the sample to react to the abrupt change in relative humidity was measured. The PMMA coated sample had the fastest response time of the three sample. This shows that using PMMA and PVA to coat tapered fiber is a viable design choice.

Keywords: Relative humidity sensors, tapered fiber, PVA, PMMA.

SURUHANJAYA PENYELESAIAN FIBER OPTICAL UNTUK PENILAIAN HUMIDITAS RELATIF

ABSTRAK

Dalam projek ini, kesan bahan salutan seperti PVA dan PMMA pada gentian tirus terhadap kepekaan kelembapan relatif dikaji. Penyikat api digunakan untuk membentuk serat menjadi serat tirus sub-panjang gelombang. Diameter sampel yang digunakan dalam eksperimen adalah dalam julat antara 2 - 10 µm. Output kuasa setiap sampel diukur sementara sensor yang dicadangkan berada dalam ruang tertutup apabila kelembapan relatif meningkat antara 35-85% RH. Sensor dengan diameter di bawah 4 µm menunjukkan kepekaan yang baik dengan resolusi halus. Sampel diameter 2.6 µm mempunyai sensitiviti tertinggi, resolusi terbaik dan sisihan piawai terkecil. Oleh itu, sampel dengan diameter 2.6 µm dipilih untuk proses salutan. Sampel bersalut PVA mempunyai kepekaan, linear dan resolusi terbaik; diikuti oleh serat terdedah dan sampel bersalut PMMA. Seterusnya, kestabilan ketiga-tiga sampel yang terdedah, bersalut PVA, dan PMMA bersalut diuji. Output kuasa setiap sampel terus diukur dalam kelembapan relatif tetap di dalam ruang terkunci. Semua bilik menunjukkan prestasi yang baik dan stabil. Walau bagaimanapun, sampel bersalut PVA adalah yang paling konsisten. Akhirnya, sambutan dan masa pemulihan ketiga-tiga sampel diukur. Setiap sampel dipindahkan di antara dua ruang dengan kelembapan relatif yang berbeza dan masa yang diambil untuk sampel bertindak balas terhadap perubahan mendadak dalam kelembapan relatif diukur. Sampel bersalut PMMA mempunyai masa tindak balas terpantas ketiga sampel. Ini menunjukkan bahawa penggunaan PMMA dan PVA untuk gentian tirus adalah pilihan reka bentuk yang berdaya maju.

Keywords: Sensor kelembapan relatif, serat tirus, PVA, PMMA.

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Thanks to God I am now able to complete my project. I am truly grateful for my family's support and love. This project would not exist without the guidance and advice of my supervisor Prof. Ir. Dr. Sulaiman Wadi Harun, who has been generous in sharing his expertise in the field of photonics.

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LIST OF SYMBOLS AND ABBREVIATIONS

2D	:	Two dimensions
3D	:	Three dimensions
ASE	:	Amplified spontaneous emission
CNT	:	Carbon Nano Tubes
DNA	:	Deoxyribonucleic Acid
FIB	:	Focused Ion Beam
FSR	:	Free Spectral Range
FOS	:	Fiber Optics Sensor
EW	:	Evanescent Wave
HEC/PVDF	:	Hydroxyethylcellulose / Polyvinylidenefluoride
MF	:	Micro Fiber
MWNTs	:	Multi Walled Nano Tubes
nm	:	Nanometer
OPM	:	Optical Power Meter
OMF	:	Optical Micro Fiber
OMR	:	Optical Micro Resonator
R	:	Radius
Si	:	Silica
SMF28	:	Single Mode Fiber 28
TIR	:	Total Internal Reflection
TLS	:	Tunable Laser Source
PVA	:	Poly Vinyl Alcohol
PMMA	:	Poly Methyl Meth Acrylate
RH	:	Relative Humidity

RI	:	Reflective Index
RPM	:	Revolution Per Minute
SMF	:	Single Mode Fiber
ZnO	:	Zinc Oxide
μ	:	Micro

CHAPTER 1: INTRODUCTION

1.1 Optical Fiber Sensors

Fiber optic cables are waveguides that carry light pulses from the source to its destination. A fiber optics cable consists of three parts: buffering for protection, cladding, and core to transmit light via total internal reflection. Silica fiber optics cable are widely used due to their low losses, small size, and flexibility (Ghatak & Thyagarajan, 1998). They are traditionally used in telecommunication. However nowadays, they are also being used in building sensors. Compared to traditional electronic sensors, fiber optics sensors are immune to electromagnetic interference and can be used in dangerous and potentially explosive environments (Thyagarajan & Ghatak, 2007).

On the other hand, relative humidity accurate measurements are vital to many industries. Thus, overcoming traditional humidity sensors limitations, such as cost, response time, and sensitivity is worthwhile (Peng, Zhao, Chen, & Xia, 2018). Optical fiber sensors are a strong candidate for replacing traditional sensors due to their small size, flexibility, low cost, stability in volatile environments. There has been extensive research to design relative humidity sensors using optical fiber. A promising direction is in the use of coated tapered microfibers. Tapering happens by flame brushing a regular fiber after stripping the buffering layer. This results in increasing the intensity of the evanescent mode propagating outside the core. So, the fiber becomes more responsive to changes in the outside environment(Peng et al., 2018).

Coating a tapered fiber increases its sensitivity to changes in relative humidity by allowing more light to escape from the tapered fiber. The coating material changes its refractive index with varying relative humidity, causing changes in the transmitted light through the core. By measuring the resulting changes in the power output, we can measure the relative humidity in the fiber's environment (Peng et al., 2018). For example in 2008, Zhang et al. used a 680 nm microfiber coated with gelatin to sense relative humidity (Zhang, Gu, Lou, Yin, & Tong, 2008). In other works, Lokman et al. used hydroxyethylcellulose / polyvinylidenefluoride (HEC/PVDF) to coat a 5 µm fiber and measure relative humidity (A. Lokman, Arof, & Harun, 2015) (Asiah Lokman et al., 2014). Most recently, Mohamed et al. coated a 6 µm fiber with multi-walled carbon nano-tubes (MWCNTs) slurry to increase humidity sensitivity (Mohamed et al., 2017).

In our work, we will construct a microfiber with diameter smaller than 10 µm and measure its power response to varying relative humidity. Then, we will coat this microfiber with PVA and PMMA and repeat the process to compare the sensitivity of the coated and bare microfiber.

1.2 Problem Statement

Optical fibers are used in many applications such as telecommunication and sensing. Research in fiber optics sensor is expanding in many directions; for example, various sensors have been developed for sensing temperature, humidity, gases, chemical materials, and medical processes. Tapered optical fibers are of special interest for sensing applications since tapering increases fiber sensitivity to its surrounding. This is a result of the enhanced power of the evanescent wave (EW) in the cladding layer. Coated microfiber has the potential to be the building blocks of smaller, more sensitive, and more stable sensors. In our work, we will attempt to construct micro fibers with diameter smaller than 10 microns. Then, coat these fibers and compare their sensitivity to changes in humidity (relative humidity).

1.3 Objectives

This work will explore the feasibility of using tapered fiber with coating as a relative humidity sensor to achieve the objectives stated below:

- To fabricate and study the behavior of bare microfiber with varying humidity.
- To successfully coat the tapered fiber with the selected coating material.
- To study the behavior of coated microfiber with varying humidity.

1.4 Report Outline

This project report consists of five chapters. The first chapter includes the project's background, its problem statement, and objectives. The literature review is presented in chapter two, which discusses fiber optics cable, sensor applications, relative humidity sensors, tapered fiber sensors, and the various coatings used to increase the sensitivity of tapered fiber sensors. Chapter three provides an explanation of the fabrication of tapered fiber, and the process of choosing the optimal diameter for the sensor. The performance of the coated tapered fiber sensors in detecting changes in relative humidity is presented in chapter four. Finally, the conclusion and future direction of this work is shown in the last chapter.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction to optical fiber

Optical fibers are thin, transparent, and flexible waveguides made of dielectric materials, usually silica or plastic. They are used in many applications, such as telecommunication and sensing (Agrawal, 2010). Figure 2.1 shows a basic structure of an optical fiber, which consists of a protective buffering, a core, and a cladding (Thyagarajan & Ghatak, 2007). Light is guided through the fiber because the core has a larger refractive index than the cladding resulting in total internal reflection (Ghatak & Thyagarajan, 1998).

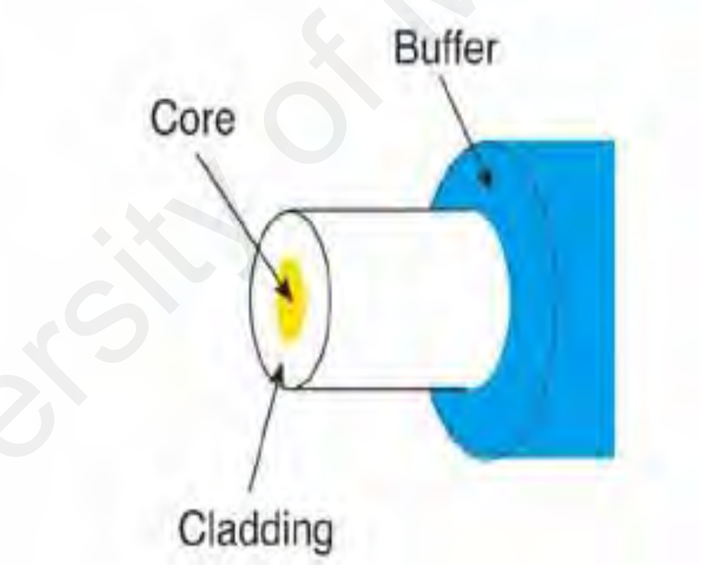

Figure 2.1: Basic structure of an optical fiber (Thyagarajan & Ghatak, 2007).

In recent years, using optical fibers for sensing is becoming more relevant. This is a result of the fiber optics advantages over traditional sensors. For example, fiber optics small size, low cost, immunity to electromagnetic interference, lower power consumption, and stability in volatile environments makes them a better option for the future of sensor fabrication (Ignacio R. Matias, Satoshi Ikezawa, 2017). Nowadays, fiber optics sensors are being developed to measure various parameters, such as temperature, humidity, gases, chemicals, and magnetic field intensity, as shown in Figure 2.2 (Ignacio R. Matias, Satoshi Ikezawa, 2017). Most of these sensors map variation in the power output to changes in the measured parameter or measure the peak wavelength shift that results from a changing parameter in the sensor's environment (Wu et al., 2017).

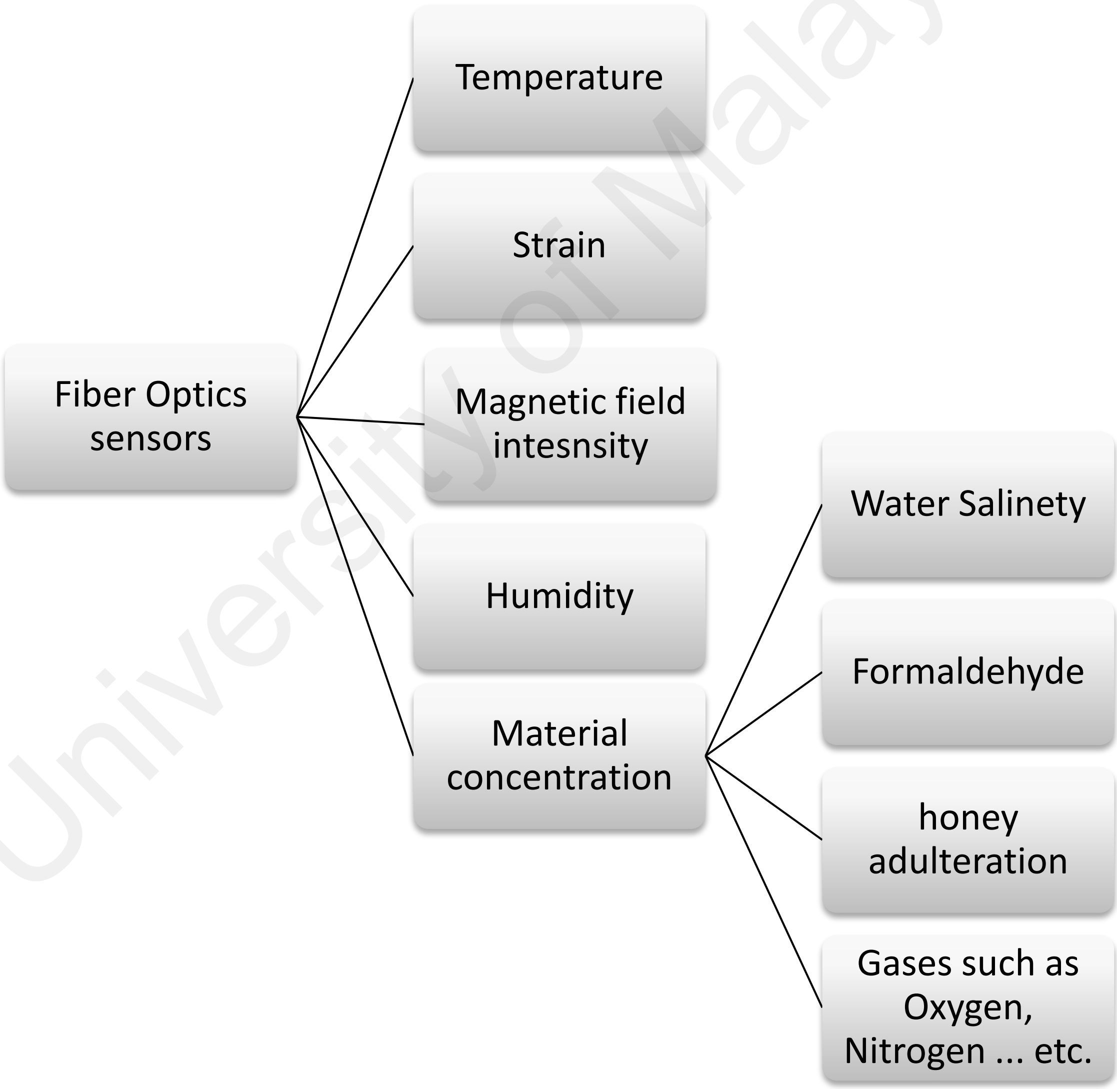

Figure 2.2: Various applications for fiber optic sensors

2.2 Fiber Optics Sensors

An optical fiber sensor is a device that uses the light propagation properties of its cable to provide reliable sensing for a parameter in its environment. Fiber based sensors are becoming more popular since traditional sensors such as electro-mechanical and thermo-mechanical sensors do not perform well in harsh environment due to corrosion, electromagnetic interference, and deformations(Colin Tong, 2010). For environmental sensing, humidity sensors need to work well for very long periods of time in harsh environment. Thus, developing sensors that satisfy these requirements is vital and optical fiber technology provides a promising candidate for these sensors.

Fiber optics sensors can be classified into point based and distributed sensors. Distributed sensors provide measurements over long distances by transmitting light through a "freestanding fiber" and measuring the back-propagating scattered light at the transmitter side. On the other hand, point based sensors produce measurements at specified locations, responding to changes in the surrounding environment of these locations (Joe, Yun, Jo, Jun, & Min, 2018). Additionally, fiber optics sensors can be classified based on their fabrication process. For instance, fiber shaping or coating material can be used to categorize sensors. Side-polishing, tapering, splicing, and machining are few of the optical shaping technique used to enhance a fiber's sensitivity, the shaping processes are demonstrated in Figure 2.3 (Joe et al., 2018). Coating fiber with graphene, metal oxides, or polymers also improves sensors performance(Joe et al., 2018).

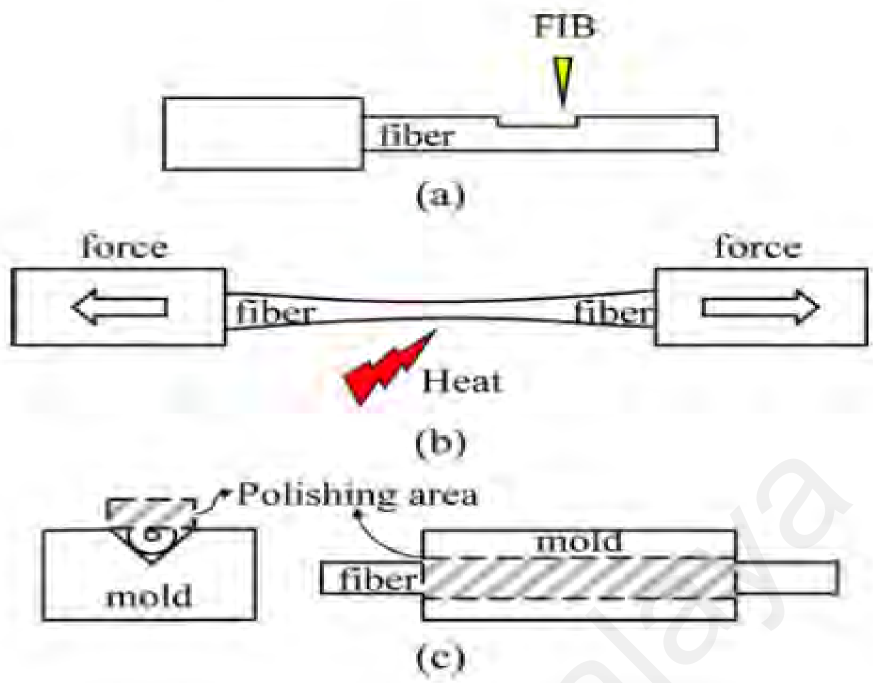

Figure 2.3: Fiber shaping methods (a) Focused ion beam direct machining, (b) tapering and (c) side-polishing (Joe et al., 2018).

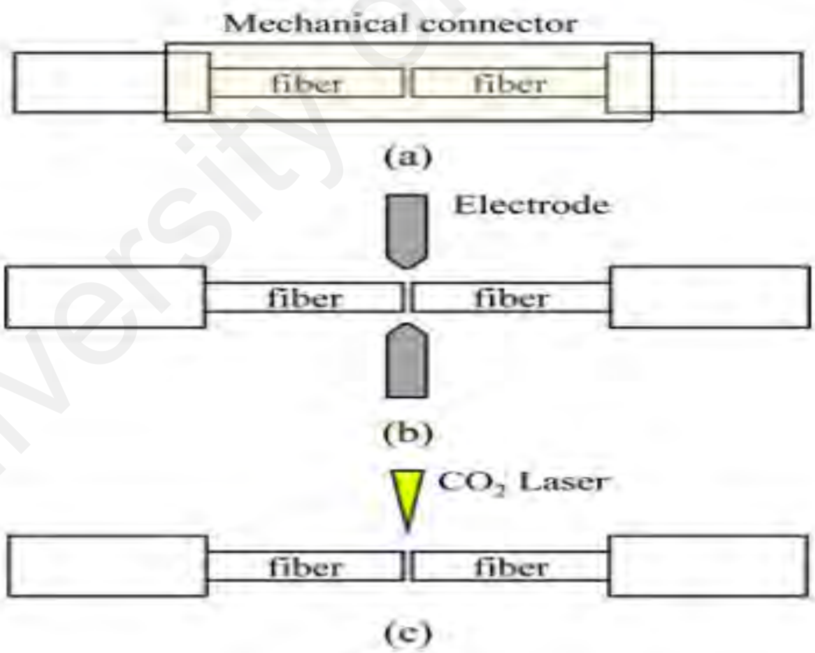

Figure 2.4: Splicing techniques (a) mechanical, (b) arc fusion, and (c) laser splicing (Joe et al., 2018).

Fiber cables need to be altered to become sensitive to its environment. Changing the fiber shape makes the light passing through the fiber more susceptible to the surrounding environment. The shaping of the fiber causes more light to be propagated outside the core of the fiber in the evanescent mode, which is highly responsive to changes in its environment. Consequently, the power output of the fiber becomes dependent on the parameters of its surroundings, which behaves now as part of the core's cladding (Krohn, MacDougall, & Mendez, 2015).

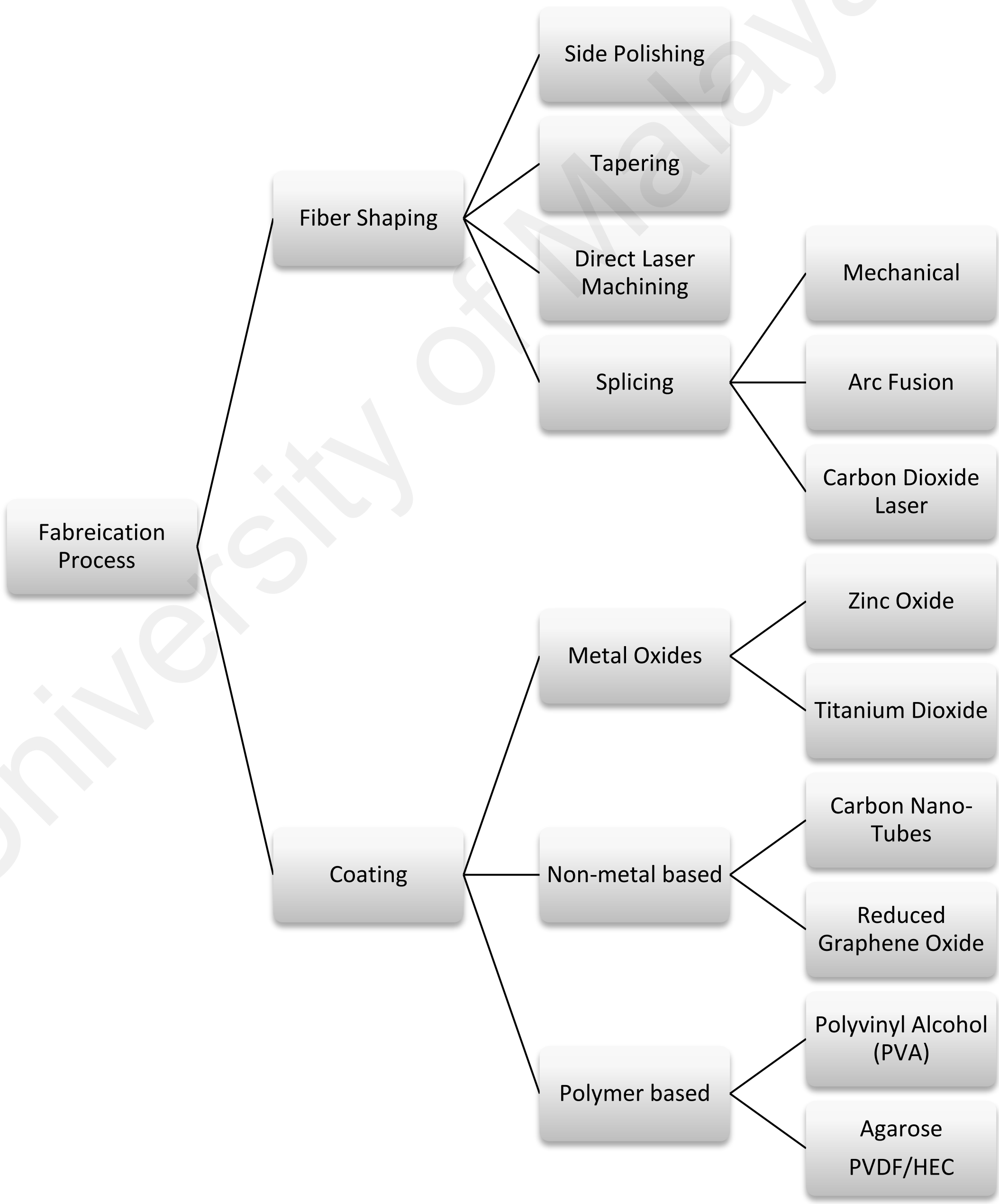

Figure 2.5: Fiber optics sensors fabrication processes.

Additionally, coating the fiber with certain materials can control and enhance this newfound sensitivity. The coating material interacts with the changing parameters of the sensor's environment. The result of this interaction is changing the refractive index of the coating that now behaves as part of the cladding. Thus, the light propagation characteristics of the sensor change with variations in its environment. (Peng et al., 2018). Some of the fabrication processes of fiber sensors are included in the graph above.

2.3 Tapered Optical Fiber

Due to fiber optic cables strength and flexibility, it is used in constructing sub-wavelength cables that are used in building, couplers, filter, and sensors. These sub-wavelength cables are usually called tapered microfibers(Chen, Li, & Xu, 2018).

A regular fiber optics cable is immune to changes in its surrounding environment due to its thick cladding compared to its core causing the evanescent mode outside the core to have a negligible effect on the transmitted power(Corres, Arregui, & Matias, 2006). Tapering is the process of reducing the diameter of the fiber gradually until the core diameter is in the sub-wavelength region. The smallest diameter region of the tapered fiber is called the waist, as shown in figure 2.6 from (Y. Tian et al., 2011).

Tapered microfibers make good sensors because they interact strongly with their environment because the evanescent mode outside the core is larger. This property made tapering a valuable method for building temperature, humidity, gases, and strain (Ignacio R. Matias, Satoshi Ikezawa, 2017).

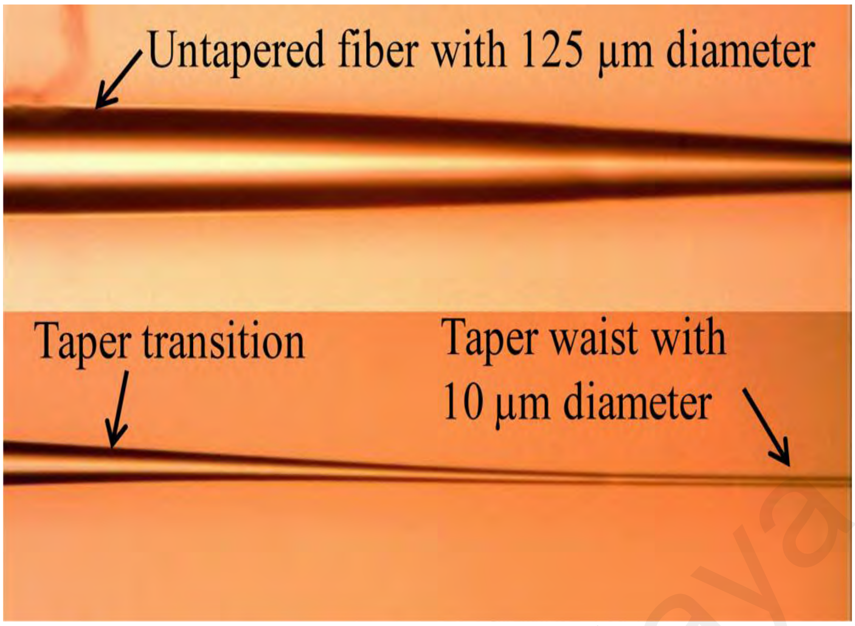

Figure 2.6: Tapered fiber regions and dimensions (Y. Tian et al., 2011).

2.3.1 Tapering Techniques

Tapering is reducing the fiber the fiber radius to cause changes in its light propagation characteristics. There are many methods to achieve tapering. For example, fiber pulling, direct drawing from bulk material, lithography, and laser ablation. Flame heating method has been proven to fabricate small diameters with good performance and physical attributes (S. W. Harun, Lim, Tio, Dimyati, & Ahmad, 2013).

Flame heating process reduces the diameters of both the core and the cladding. The smallest diameter region is called the waist. Between the waist and the regular size fiber is a transition region in which the diameter of the core and the cladding is steadily decreasing (S., W., Arof, & Ahm, 2012). Flame heating is usually done by heating the fiber with a flame while pulling on its ends to reach a certain diameter (Brambilla, 2016). An example of the rig used to achieved tapering from (S. et al., 2012) is shown in figure 2.7.

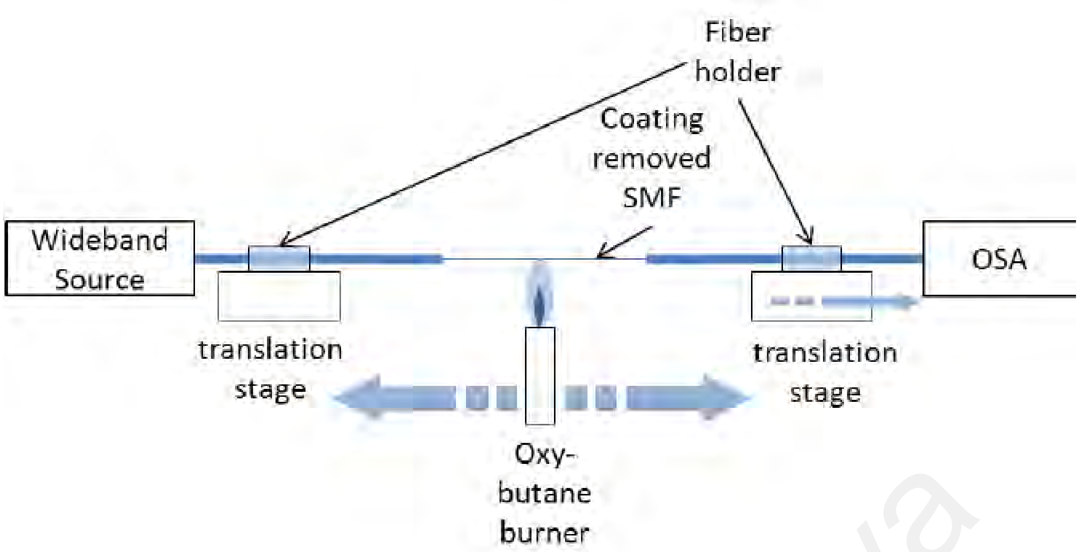

Figure 2.7: Flame brushing rig for tapering fiber (S. et al., 2012).

2.4 Tapered Fiber Sensors

When a tapered fiber's diameter becomes smaller, a larger fraction of the light propagates in the cladding's evanescent field, as shown in figure 2.8 from (Garcia-Fernandez et al., 2011). This makes the fiber highly responsive to any changes in its surrounding environment. The increased sensitivity of the tapered fiber makes it perfect for building sensors (Brambilla, 2016).

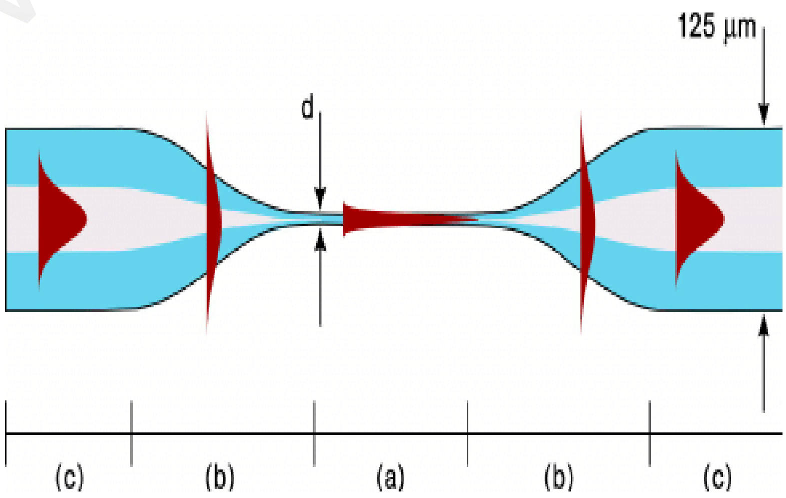

Figure 2.8: Evanescent mode in tapered fiber (Garcia-Fernandez et al., 2011).

When a single mode fiber is tapered by flame brushing for instance, the core cladding diameters shrink and their interface change. The tapered region of the fiber starts behaving as a multimode fiber. The light from the tapered waist travels through the entire fiber and the outside environment acts as a cladding. As a result, interactions between the environment and the light are stronger and any small changes in the environment are directly reflected in the transmitted power of the cable. This is the principle of work to many optical fiber sensors (Corres et al., 2006; S. W. Harun et al., 2013).

2.5 Relative Humidity Sensors

Relative humidity is the ratio between water vapor in the air to its amount in saturated air at a certain temperature (Yeo, Sun, & Grattan, 2008). Measuring relative humidity is vital to many fields. Health services, textile industry, food processing, agricultural facilities and many other field need accurate, stable, and inexpensive sensors for relative humidity (Peng et al., 2018). For example, even the smallest traces of water in the materials used to produce semiconductors can severely degrade the quality of the final product (Wang & Wolfbeis, 2016). Thus, monitoring relative humidity is crucial to electronics manufacturing and many other products. Examples of humidity sensing application from (Yeo et al., 2008) is shown in figure 2.9.

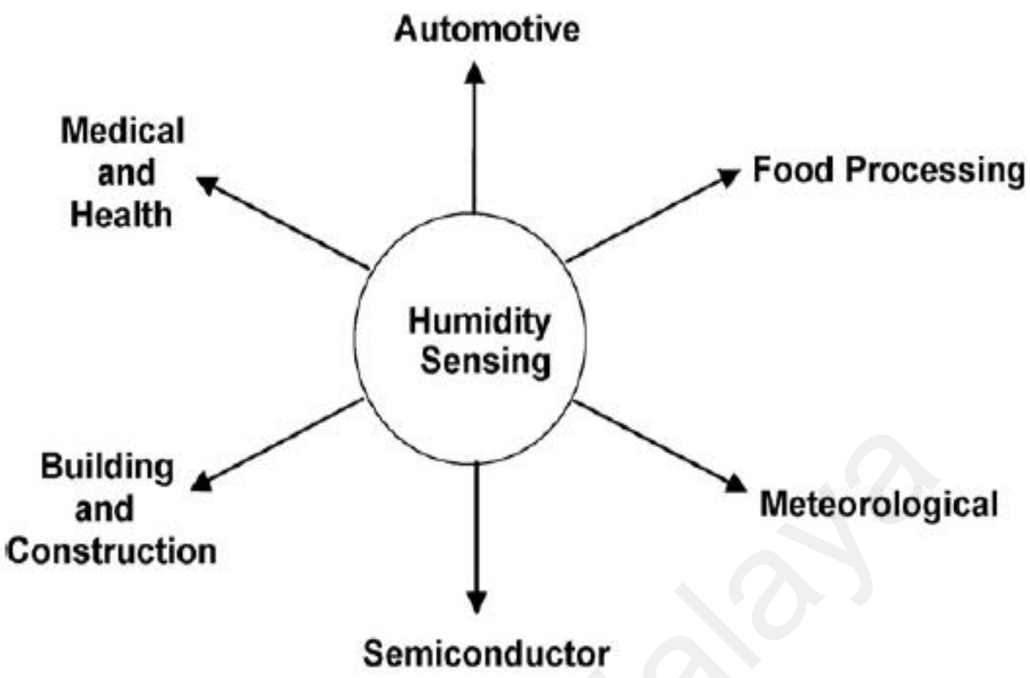

Figure 2.9: Application of humidity sensing (Yeo et al., 2008).

Conventional relative humidity sensors are of limited use due to their size, cost, and to electromagnetic interference. Thus, fiber sensors provide a suitable alternative since fiber optics are immune to electromagnetic interference, works well in volatile environment, and stable (Peng et al., 2018).

Optical fiber relative humidity sensors can be classified based on their structure to tapered, fiber grating, coupler, resonator, and interferometer relative humidity sensors. This classifications highlights the great potential that fiber optics have for building relative humidity sensors(Peng et al., 2018). In this project, only tapering will be used to build relative humidity sensors. Additionally, relative humidity fiber sensors can be categorized based on their coating material. The following sections will discuss this in details.

2.5.1 Tapered fiber humidity sensors

Tapering lowers the core and cladding diameter and increases the effect of evanescent mode. Tapering fiber is a relatively simple procedure that can greatly increase regular fiber optics sensitivity to relative humidity. Both plastic and silica fiber can be tapered to build relative humidity sensors. Tapering will be discussed in details in the next chapter.

2.5.2 Fiber Grating relative humidity sensors

Fiber gratings are periodically placed region of different refractive index inside the fiber intruding controlled scattering that reflects back specific wavelengths. If the periods of the gratings are less than $1\mu m$, the fiber is called fiber Bragg gratings. If the periods are between 10 to $100\mu m$, the fiber is called long gratings fiber (Hecht, 2017). Micro fiber Bragg gratings have been used in designing relative humidity sensors by chemical etching. The changing refractive index inside the gratings and in the air around the fiber makes it responsive to changing relative humidity. Thus, relative humidity is measured by measuring the Bragg wavelength, for example (Shao et al., 2015) reported a sensitivity of 3 pm/%RH between 50-80%RH.

2.5.3 Coupler relative humidity sensors

Micro fiber couplers are made by fusing two fibers together by heat brushing. The two evanescent modes that result from the fusion and tapering respond strongly to changes in relative humidity, especially if the coupler is coated by any material that changes its refractive index when relative humidity changes (Peng et al., 2018; Yeo et al., 2008). The center dip wavelength changes when relative humidity varies. (Bo, Wang, Semenova, & Farrell, 2015) obtained a sensitivity of 2.23 nm/%RH using this method with polyethylene oxide coating.

2.5.4 Resonator relative humidity sensors

Creating a self-touching structure in the tapered region of a micro fiber makes a resonator. Consequently, the wave passing through the resonator is split into two parts. The first wave propagates through the coil and the second via self-coupling (Ignacio R. Matias, Satoshi Ikezawa, 2017). Resonators can be classified into micro fiber loop resonators or micro fiber knot resonators. The resonance wavelength of the resonator changes when relative humidity change due to refractive index dependence on the shape and structure of the fiber. For instance, (Jali et al., 2019) obtained a sensitivity of 0.2053 dB m/%RH using microfiber loop resonator of 7μm diameter.

2.5.5 Interferometer relative humidity sensors

Interferometers pass light of the same frequency and phase into different paths; then reunite then and measure the spectrum of the light output. The center wavelength shift indicates changes in the sensors environment. The evanescent mode of tapered interferometer reacts to changes in relative humidity. (Soltanian et al., 2016) obtained a sensitivity of 0.02 nm/%RH using a micro fiber interferometer.

2.6 Coating Material for Relative Humidity Sensors.

After tapering a fiber to a sub-wavelength diameter, the power output becomes more dependent on changes in the environment. However, to increase this ability coating materials are used. These coatings change their refractive index when relative humidity varies. Consequently, converting some of the guided light through the fiber to radiation outside the fiber, causing a power output change depending on relative humidity variations (Yeo et al., 2008). In the following section, we will discuss few of the materials that have been used to coat tapered fiber relative humidity sensors.

2.6.1 Metal Oxide (Zinc-Oxide)

The porous nature of metal oxides, for example Zinc-oxide, diffuses water vapor in the coating layer (Peng et al., 2018). The water in the air is absorbed by the porous layer when its pressure exceeds the pressure of the saturated water in the oxide layer. Otherwise water is leaked from the oxide layer. The shape and size of the pores controls the performance of this process(Mohamed et al., 2017).

For zinc-oxide, the absorption of water increases with higher relative humidity. Water-filled pores will have a higher refractive index. Causing a change in the propagation characteristics of light through the sensor depending on relative humidity(Liu et al., 2012).

2.6.2 Carbon Nano-tubes

Carbon nano-tubes are infinitely extended cylinder of one or several graphite sheets (Peng et al., 2018). Depending on their orientation and diameters these nano-tubes have metallic or semi-conductive properties. Multi-walled Carbon nano-tubes have good optical properties due to the van hove singularities. Additionally Multi-walled Carbon nano-tubes show great strength and tensile elasticity (Komatsu, 2010). Multi-walled Carbon nano-tubes are constructed by using thermo-chemical deposition. This coating increases tapered fiber sensitivity to relative humidity due to the drastic variations in the refractive index when this material absorbs water from its surrounding atmosphere(Mohamed et al., 2017).

2.6.3 Agarose Gel Coating

Agarose is a biopolymer gel mainly used in separating DNA chains(Righetti, 2016). Agarose is a white powder soluble in water at certain temperature. The resulting gel can be used as a coating for relative humidity sensors to increase sensitivity. Due to its porous nature, it is capable of absorbing water from the atmosphere, changing the gel's refractive index and the sensors output power (Batumalay et al., 2014).

2.6.4 Hydroxyethylcellulose/Polyvinylidenefluoride(HEC/PVDF) Composite

A combination of HEC/PVDF has several advantages such as cost and availability (Xia, Li, Li, Kou, & Liu, 2013). With proper and careful dissolving a 3D mesh gel can be obtained from this combination. This gel absorbs water from the surrounding environment changing its refractive index and altering the power output through the sensor depending on the environments' relative humidity(A. Lokman et al., 2015).

2.6.5 Polyvinyl Alcohol (PVA) Composite

PVA is a strong hydrophilic polymer that is soluble in water. PVA can absorb several times its volume in water. Additionally, PVA adheres to silica well, making it a good choice for thin films (Rong et al., 2013). When PVA coated tapered sensor experiences changes in relative humidity, PVA changes its properties, such as swelling degree, and refractive index. Consequently, this affects the light propagation in the sensor and the power output of the sensor. Thus, tapered fiber with PVA coating can be used to sense relative humidity changes (Chan et al., 2012).

2.6.6 Poly (methyl methacrylate) (PMMA)

PMMA is a synthetic polymer that shows chemical durability and resistance to ultraviolet light. Most importantly, PMMA refractive index changes with changing state of its surrounding (Ali, Karim, & Buang, 2015). For example, changing relative humidity around PMMA coated tapered fiber results in PMMA layer increasing its refractive index. Thus, increasing the power loss in the coated fiber. (Irawati et al., 2017)

2.7 Sensing Relative Humidity Using Coated Tapered Fiber

There have been several attempts to measure relative humidity using coated tapered fiber. For example, in 2008, (Zhang et al., 2008) constructed a 6.8µm fiber coated with 0.88µm layer of gelatin. Increasing relative humidity in the surroundings of the tapered region diffuses water into the gelatin layer changing its refractive index. Consequently, transforming part of the guided mode into radiation mode and increasing loss with increasing relative humidity. This senor operated in the 9% to94% range with 0.118 dBm/RH% on average and had a response time of 70ms.

(Irawati et al., 2017) constructed two sensors for relative humidity. One was silica and the other plastic fiber. They were coated with zinc oxide and the voltage output was recorded while varying relative humidity. The plastic-based sensor performed better than the silica-based with sensitivity of 0.176mV/RH% and linearity of 94.41%.

In 2014, (Asiah Lokman et al., 2014) proposed a sensor using HEC/PVDF coating on a 5 µm tapered fiber. The polymer's reaction to changes in relative humidity improves the tapered fiber sensitivity to relative humidity. This sensor had a sensitivity of 0.0228dBm/RH% and slope linearity larger than 99.91% in the 50%-80% RH range.

Recently, (Mohamed et al., 2017) demonstrated a relative humidity sensor that used a multi-walled carbon nano-tubes. A 6 µm tapered fiber was enclosed in the carbon slurry. This resulted in increasing the sensitivity from 3.811mW/RH% for bare fiber to 5.71mW/RH% for the slurry coated one in the relative humidity range of 45% to 80%.

CHAPTER 3: TAPERING OF FIBER FOR HUMIDITY SENSING

3.1 Introduction

There is a growing interest in using tapered micro fiber to build environmental sensors. Tapering is the process of using a chemical, mechanical or thermal method to lower the diameter of an optical fiber (Chen et al., 2018). This results in increased sensitivity to changes in the environment of the tapered fiber caused by the evanescent mode propagation. When a fiber is tapered, a portion of the light that usually travels through the core radiates into the outside of the fiber creating the evanescent mode and a larger power loss compared to ordinary fiber (Yeo et al., 2008). The tapered region of single mode fiber behaves like a multimode fiber since light is flowing through the cladding layer. Thus, the surrounding environment of the fiber works as the cladding of the tapered region. This mode coupling in the tapered waist of single mode fiber makes it very responsive to changes in the refractive index of its surroundings. The light guided through the cladding of the tapered fiber and affected by the changes in surrounding environment is called evanescent mode(Ignacio R. Matias, Satoshi Ikezawa, 2017).

The evanescent mode in tapered fiber makes it suitable for sensing applications, for example, sensing temperature, humidity, gases, and magnetic fields (Ignacio R. Matias, Satoshi Ikezawa, 2017). The evanescent mode is greatly affected by changes in the fiber surroundings. Thus, changes in the fiber environments cause changes in the propagation characteristics of the fiber, such as power loss and peak wavelength (Ascorbe, Corres, Arregui, & Matias, 2017). This is the working principle of the sensors constructed for this project.

In this project, the increased evanescent mode with smaller diameters will be used to measure humidity. Since tapering causes more light to radiate in the evanescent mode, the power output of tapered fiber is highly sensitive to changes in the surrounding environment. When the relative humidity surrounding the tapered fiber increases, the refractive index of the air surrounding, the tapered region changes. Additionally, the water particles in the air increase the scattering in the air directly near the tapered region. Consequently, the power loss of the tapered fiber is directly influenced by this change in humidity. With increasing relative humidity in the fiber environment, the power loss of the fiber increases, so measuring the power output of the fiber provides insight to relative humidity in the environment (Asiah Lokman et al., 2014).

In this chapter, the tapering method used and its effects on propagation characteristics will be discussed. Additionally, the experimental setup will be explained. Finally, a comparison between the propagation characteristics of several tapering diameters with varying relative humidity will be presented.

3.2 Fabrication of Tapered Fiber

Flame brushing was used to taper the fiber. The setup for the process consisted of two holders for the fiber, a slider stage, two stepper motors, a microcontroller board as figure 3.1 from (S. W. Harun et al., 2013) shows. The flame is made using butane gas and shaped by oxygen. Two cylinders supply the gas into a combining chamber that can ignite a flame using a regular lighter. The oxygen and butane gas is kept at 5 psi pressure, and the flow of air from the flame and the temperature are balanced to maintain a small but hot enough a flame to heat and then soften the silica fiber. The motors control the flame and one end of the fiber holder to both move the flame across a small region of the exposed fiber and to extend the stretching fiber (S. W. Harun et al., 2013).

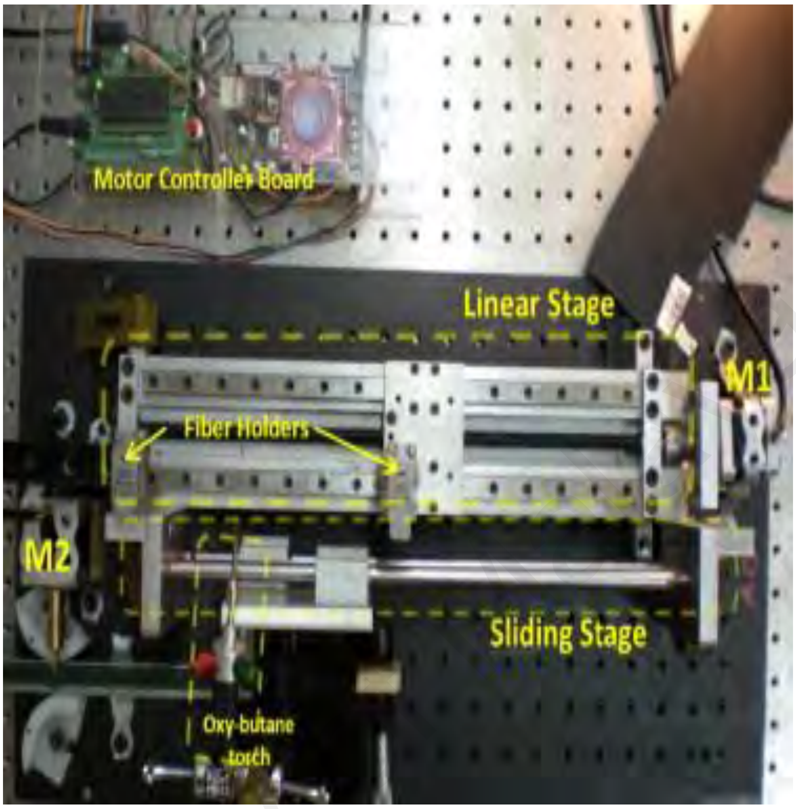

Figure 3.1: Flame brushing machine (S. W. Harun et al., 2013).

To prepare the tapered fiber, we use single mode fiber with 128 μm diameters (SMF, Corning, 128). The protective buffer is removed using a fiber stripper. Then, the exposed fiber is wiped clean using a tissue and iso-propanol solution to guarantee the removal of the buffering. The clean fiber is placed on the fiber holder to proceed with the tapering process. The stripped portion of the fiber is fixed on the holder; the flame will be moved across a small region of the fiber repeatedly by one stage motor. The other motor will stretch one end of the fiber to lower its diameter. As a result, the heat will soften the fiber and the stretching movement will cause the fiber to extend and its diameter to decrease. After completing the tapering, a microscope is used to ensure that the required diameter is reached (S. et al., 2012).

3.3 Experimental setup

To measure the tapering effect on relative humidity sensitivity, we used the setup described below. Firstly, the tapered fiber is spliced into a patch cord. This patch cord is connected to an optical power meter on one end and an ASE (amplified spontaneous emission) light source, with center wavelength of $1550\mu m$, on the other end. Secondly, the tapered region of the sample is enclosed in a locked chamber in which humidity is steadily increasing using saturated salts (Sodium Hydroxide). Additionally, the relative humidity in the chamber is measured using an electronic humidity sensor for reference. The setup is shown in figure 3.2.

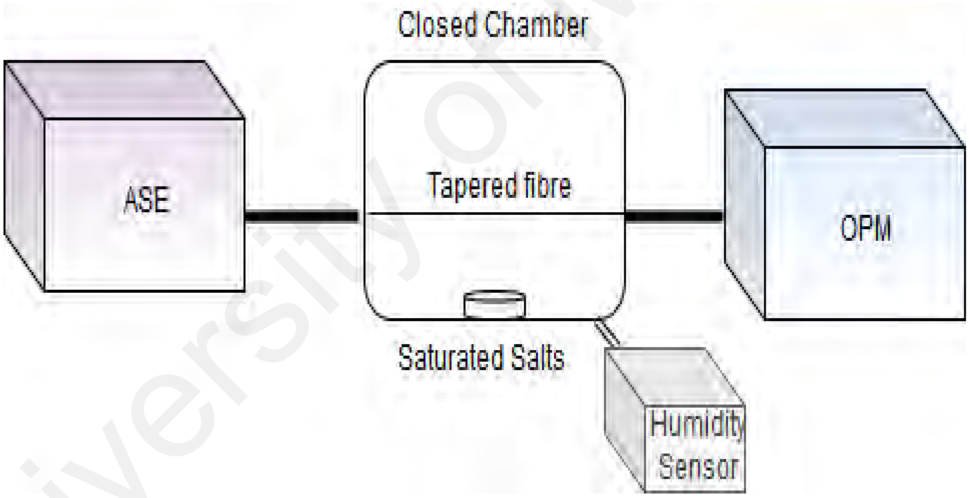

Figure 3.2: The experiment setup for bare fiber testing

3.4 Results and Discussion

The results of several samples of tapered fiber are presented in figure 3.3. Based on this collected data, a summarized analysis was included in table 3.1. It is clear that with increasing relative humidity, power loss increases as well. This loss is caused by additional scattering losses introduced with increasing relative humidity. The water particles in the chamber change the refractive index of the surroundings of the tapered region. Thus, more light travels in the evanescent mode and the power output of the sensor is decreased, making the power output of the tapered fiber a reflection of the relative humidity of its environment.(Peng et al., 2018)

In figure 3.3, the power output of each tapered sample was recorded while varying the relative humidity from 35 % to 85% in the sealed chamber. The slope of the linear fit of each samples power output gives us the sensitivity of each sample to relative humidity. Figure 3.3 shows that the tapered fibers with diameter below 4 μm have greater sensitivity with peak sensitivity at 2.6μm.

With increasing relative humidity in the sealed chamber, the absorption losses increase due to the water particles effect on the refractive index of the cladding. The tapered fiber with diameters below 4 μm have larger sensitivity due to the larger effect of the evanescent mode that makes the sensor more susceptible to changes in its environment (Jali et al., 2019; Peng et al., 2018). As the diameter of the fiber gets smaller with tapering, more light propagates in the evanescent mode outside the core. Consequently, the smaller diameters are more affected by changes in the environment. The surrounding air around the tapered waist acts as the cladding layer for the tapered fiber. Thus, changes in the composition of that air, i.e. increased relative humidity, causes its refractive index to change; resulting in direct changes to light propagation characteristics of the fiber.

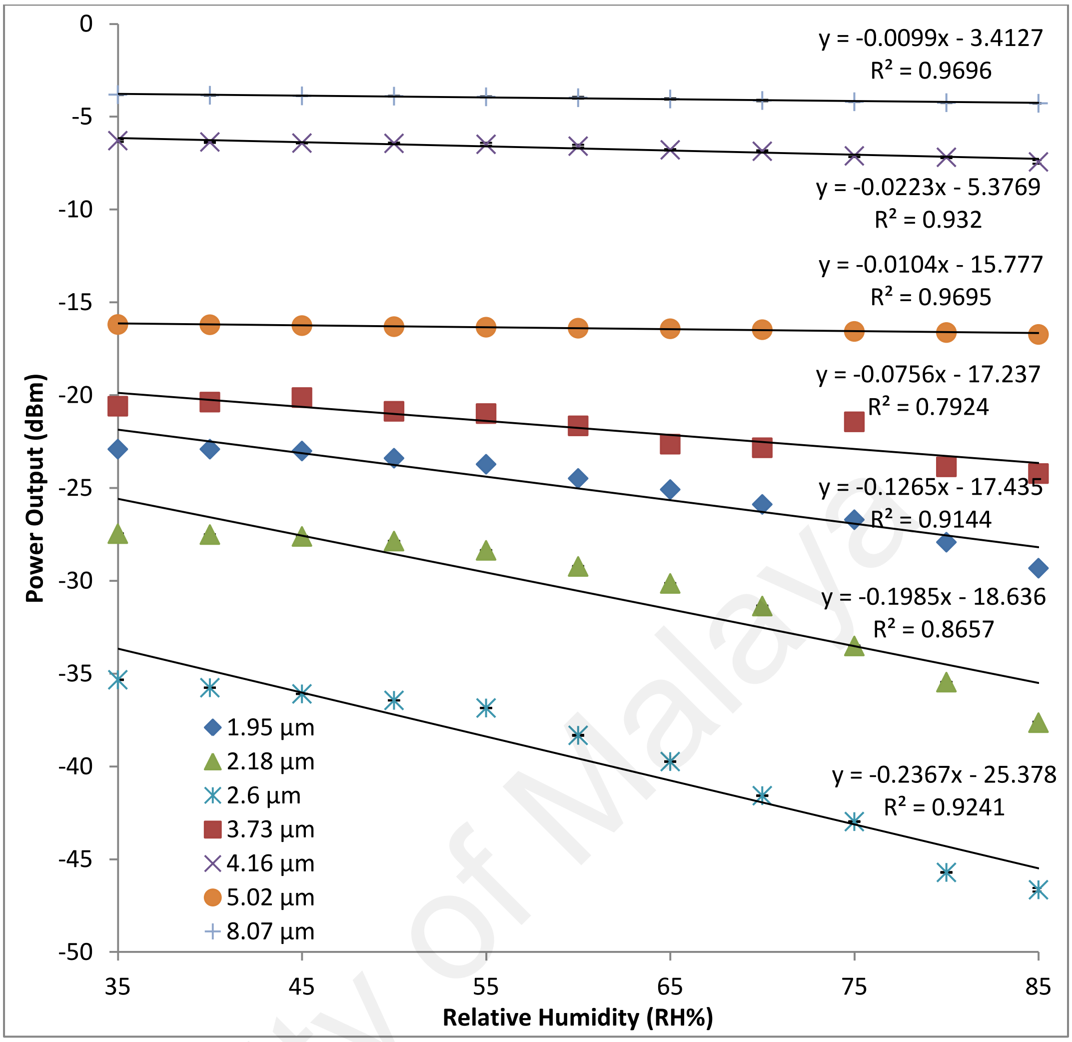

Figure 3.3: The Power output of various tapering diameters with varying humidity.

The bar chart in figure 3.4 shows that the sensitivity increases with diameters below $4\mu m$. The highest sensitivity of 0.2367 dB/ %RH achieved was at 2.6 μm diameter. Additionally, fibers with diameters below 4 μm perform much better than samples with larger diameters. This shows that tapering fiber increases its sensitivity to changes in the relative humidity of its environment. With larger waist diameters, the evanescent mode becomes weaker and the sensor is less affected by changes in relative humidity in its environment.

The increased sensitivity with diameters below 4 μm is caused by the stronger evanescent mode in these diameters. Smaller diameter for tapered fiber forces more light to propagate in evanescent mode, resulting in increased sensitivity to changes in relative humidity. However, this improvement peaks at diameters between 4 μm and 2 μm. For diameters below 2 μm, the structural integrity of the tapered region hinders its performance.

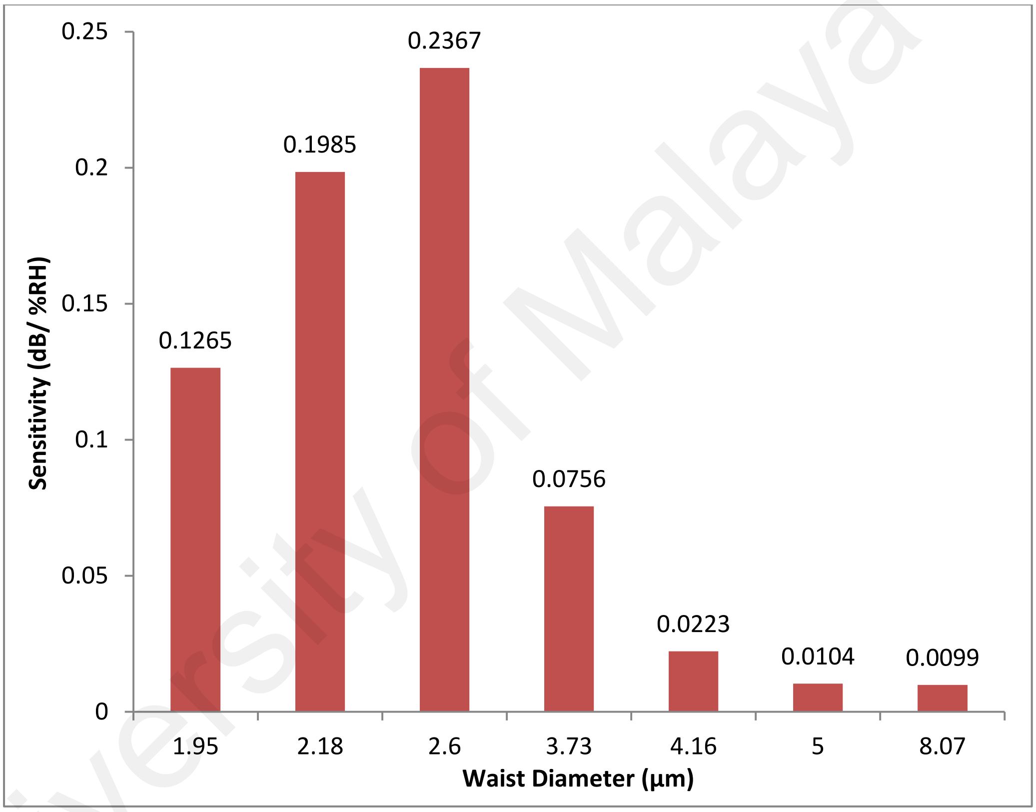

Figure 3.4: The sensitivity to relative humidity variation of various tapering diameters.

Table 3.1 shows a comprehensive comparison of the tested samples sensitivity, linearity, standard deviation, and resolution. Standard deviation was calculated from repeating each experiment for each sample three times. Resolution is simply the standard deviation of the each sample's trials divided by its sensitivity. Resolution for the samples above 4 µm is several times lower than the samples below 4 µm due to its increased sensitivity. The sample of 2.6 µm has the finest resolution and samples below

4 μm have resolution well below 1 %RH. All the samples below 4 μm showed good linearity.

However, the 2.6 μm was selected due to having a linearity of 96% and the most preferable resolution and sensitivity. This selected sample size was used in the coating stage of the experiment to study the effectiveness of the coating materials.

Table 3.1: Performance of various diameters of tapering fiber with varying relative humidity.

Diameter (μm)	1.95	2.18	2.60	3.73	4.16	5.00	8.07
Sensitivity (dBm/ %RH)	0.1265	0.1985	0.2367	0.0756	0.0223	0.0104	0.0099
Linearity (100%)	95.62	93.04	96.13	89.02	96.54	98.46	98.47
Standard deviation (dBm)	0.0156	0.0217	0.0223	0.0499	0.0562	0.0671	0.0990
Resolution (%RH)	0.1235	0.1095	0.0942	0.6604	2.5180	6.4495	10.0022
Range (% RH)	35-85	35-85	35-85	35-85	35-85	35-85	35-85

3.5 Summary

In this chapter, the tapering method and experiment setup were presented. The performance of samples with diameters below 10 μm was measured with varying relative humidity. After that, we compared the performance of our samples. Samples below 4 μm had the largest sensitivity and finest resolution and the sample of 2.6 μm diameters being the most sensitive to changing relative humidity with the best resolution of the tested samples.

CHAPTER 4: TAPERED FIBER COATED WITH PVA AND PMMA FOR RELATIVE HUMIDITY MEASUREMENTS

4.1 Introduction

Tapering increases fiber sensors ability to measure relative humidity, as was shown in the previous chapter. This sensitivity can be improved by coating the tapered region with materials that react to changes in relative humidity. The coating layer act as a new cladding layer for the tapered region since its original cladding is severely diminished after flame brushing. Thus, any changes in coatings refractive index affects the evanescent mode propagation and the power loss of the fiber (Ascorbe et al., 2017). In our work, we choose PVA and PMMA due to their high sensitivity to relative humidity. The refractive index of both of PVA and PMMA changes when relative humidity increases. As a result, the propagation characteristics of the coated fiber vary with changing relative humidity (Irawati et al., 2017; Rong et al., 2013).

In this chapter, a comparison between the performance of PVA and PMMA coated fiber is provided. The power output of a PVA coated micro fiber was measured while we varied the relative humidity. Also, we repeated this procedure for a PMMA coated sample as well. Finally, we compare the sensitivity to changes in relative humidity for both of the coated sample with a bare fiber sample to show the improvement in sensitivity caused by coating.

4.2 Coating of tapered fiber with PMMA

We choose a tapered fiber (or microfiber) with a waist diameter of 2.6 μm since it had the highest sensitivity to relative humidity to coat with PMMA. For the coating layer, we used 1 mg of crystal PMMA with 10 ml of iso-propanol. These materials were mixed and heated at 100 C° with 700 rpm for an entire hour on a hot plate. The resulting solution was carefully dripped onto our sample. The coated sample was left to dry for 24 hours (Sulaiman Wadi Harun, Yusoff, Irawati, Rahman, & Isa, 2018).

4.3 Coating of tapered fiber with PVA

To fabricate PVA 5 mg of PVA crystals have been mixed with 12 ml of distilled water. Afterwards, the mixture was cleaned in an ultrasonic bath for 6 hours at 20° C. The resultant solution was carefully dripped onto the selected sample's tapered region and left to dry for an hour.

4.4 Humidity sensing experiment

Firstly, we prepared three samples with diameter of 2.6 μm using the flame brushing method explained in the previous chapter. Secondly we use one sample and connect to a patch cord on both ends using a splicer. After that, the sample's tapered region is put into a closed chamber that we will increase the relative humidity in using saturated salts. The patch cord had light of peak wavelength 1550 μm flowing through it because we connected one end to an ASE light source. The power output was recorded using the OPM connected to the other end of the patch cord. The relative humidity inside the chamber will be monitored using an electronic humidity sensor for reference. The experiment setup is shown in figure 4.1.

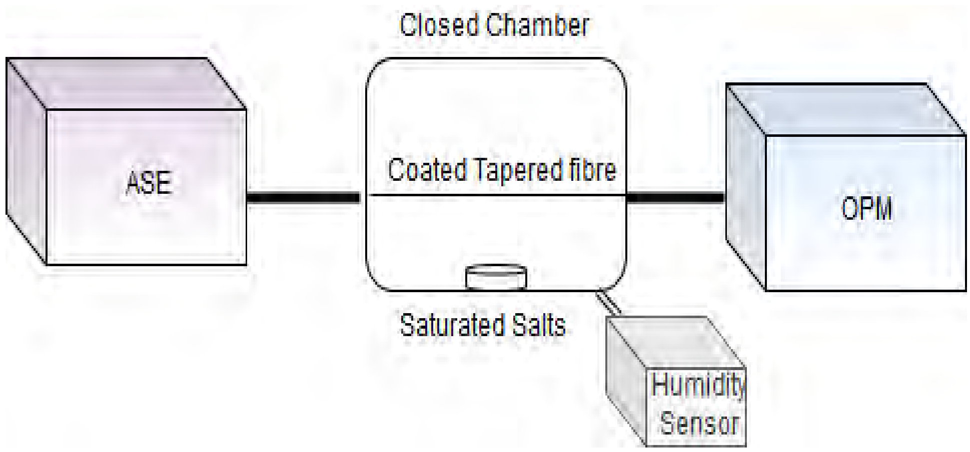

Figure 4.1: Experiment setup for testing the PVA coated and PMMA coated tapered fiber.

In figures 4.2, 4.3, and 4.4, the power output of the 2.6 μm tapered samples before and after coating with PVA and PMMA is presented. For each step, we recorded the power output of the sensor as the relative humidity was gradually increasing in the closed chamber. Each sample was tested three times in humidity range from 35-85%RH.

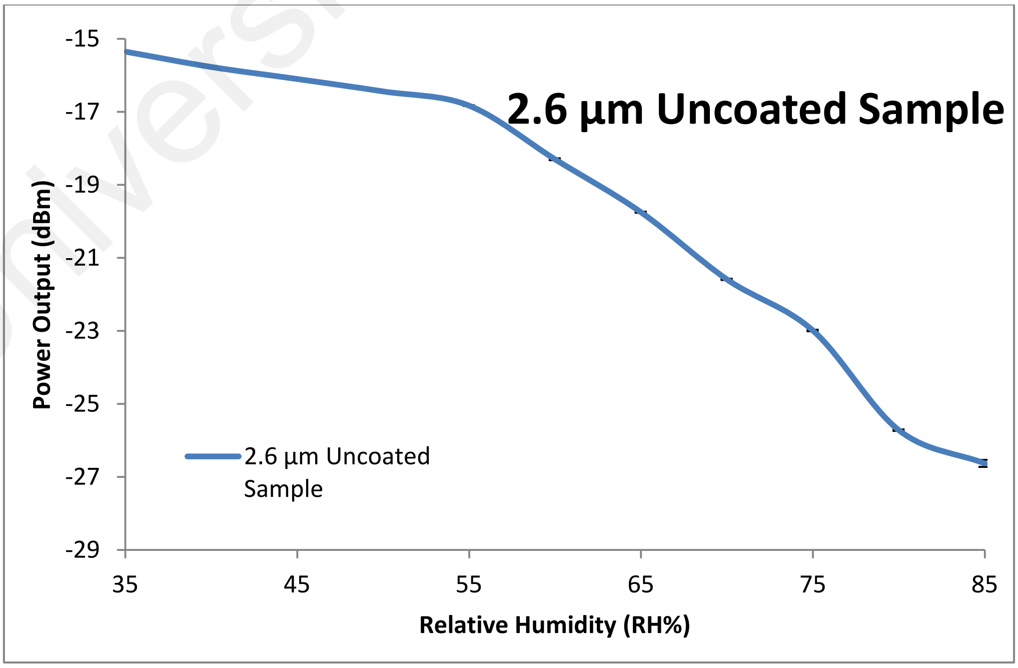

Figure 4.2: The power output of uncoated tapered fiber with 2.6 µm diameter with varying humidity.

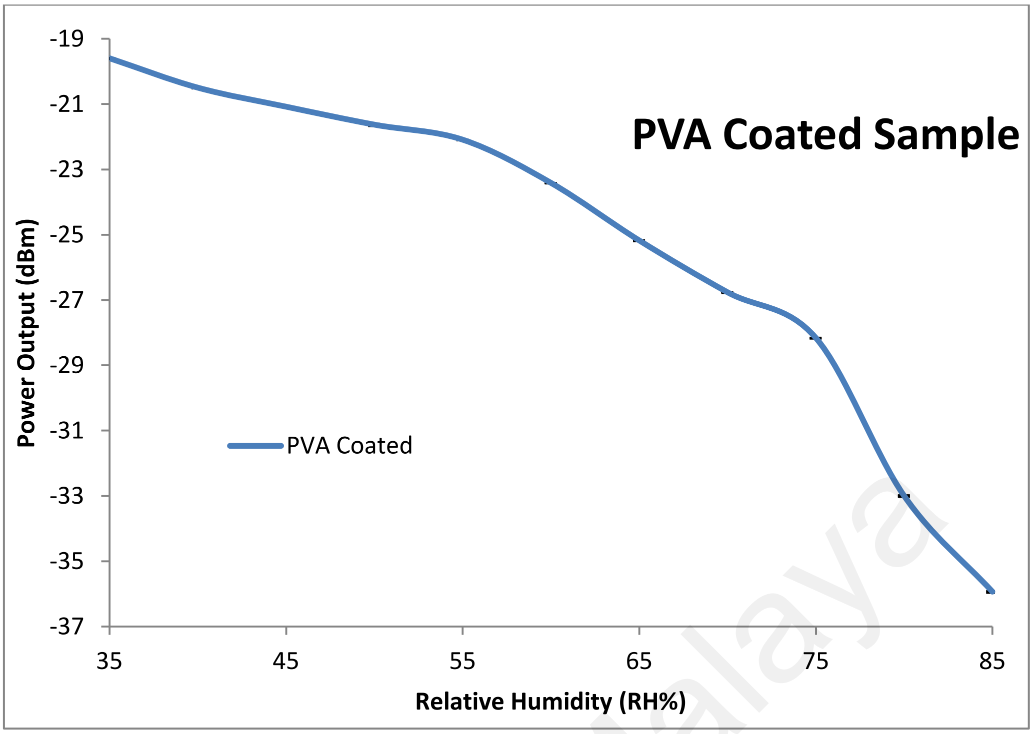

Figure 4.3: The power output of PVA coated tapered fiber with 2.6 µm diameter with varying humidity.

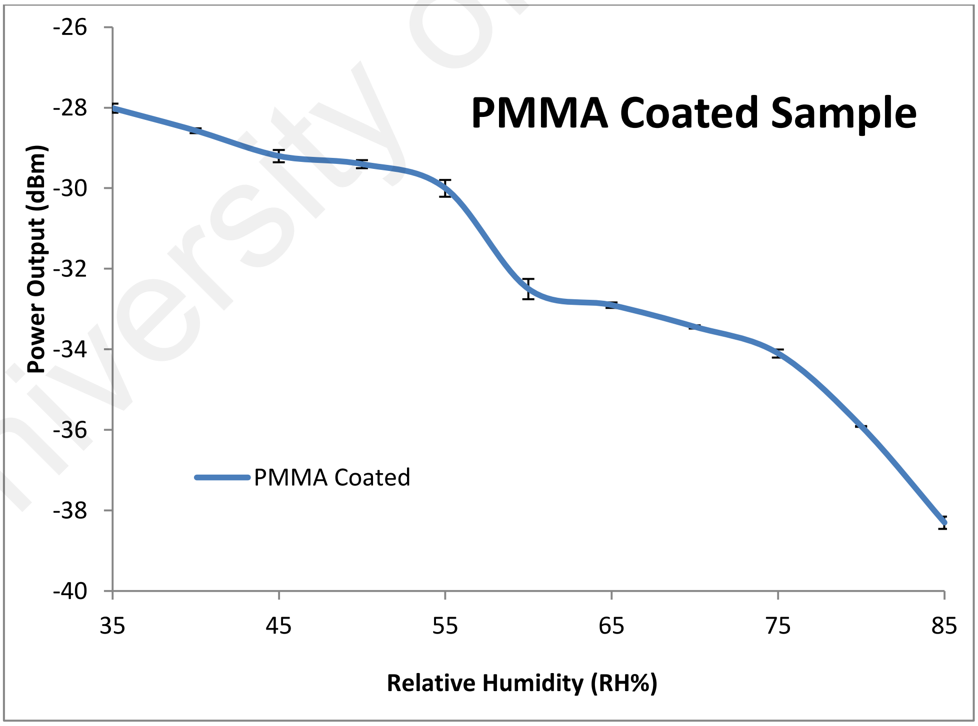

Figure 4.4: The power output of PMMA coated tapered fiber with 2.6 µm diameter with varying humidity.

From the graphs above, we can conclude that the power output decreases with increasing humidity whether the tapered fiber was coated or not. However, the PVA coating increases the total power loss by more than 3dBm and the PMMA coating introduces even a larger loss of more than 12 dBm.

The analysis of the three samples is presented in figure 4.5. Firstly, using coating lowers the overall power output regardless of the relative humidity. This is due to the tension, weight of the coating on the fiber and insertion los of the coated fiber. Secondly, the PVA coated sample showed an increased sensitivity of 0.3023 dB/RH% compared to the bare sample's 0.2367 dB/RH%. This shows the effectiveness of PVA coating tapered fiber in sensing relative humidity. The PVA coat as a new cladding layer for the tapered region. The increasing humidity in the chamber changes the refractive index of the cladding. Thus, the loss of the tapered fiber increases with humidity. The PVA coating reacts more strongly to changes in the relative humidity than air so the sensitivity of PVA coated fiber is higher (Jiao, Zhao, & Gu, 2018).

On the other hand, PMMA coating shows a lower sensitivity to changes in relative humidity than both bare and PVA coated fiber. The PMMA coated fiber has a sensitivity 0.1936dB/RH%, which is lower than the sensitivity of the bare fiber and of the PVA coated fiber. To sum up, using PVA coated tapered fiber is the most effective approach to build relative humidity sensors compared to PMMA or bare fiber.

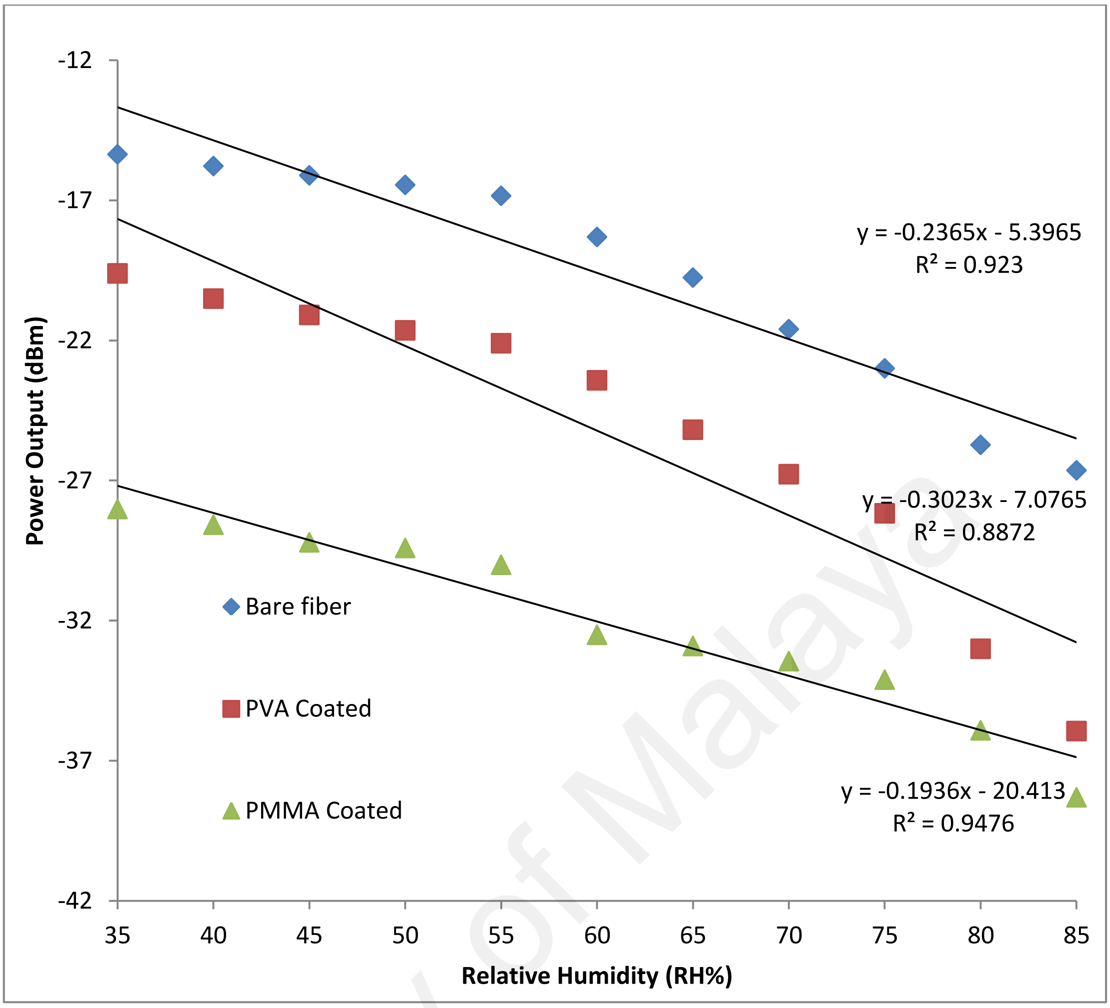

Figure 4.5: The Transmitted power levels for tapered fiber with varying humidity.

Additionally, table 4.1 compares the performance of the three samples. We see that the uncoated fiber has a slightly higher linearity than the PVA and PMMA coated samples. All the methods have linearity larger than 90%, and the experiment was conducted with relative humidity between 35-85 %.

Resolution of the PVA coated sample is the best out of the three samples we tested. While all the samples, bare, PVA, and PMMA coated have fine resolution below 1 %RH, the PMMA coated sample showed the least promising performance with resolution of 0.5 %RH. PVA coating improves the resolution of the sensor by a factor of two. Finally, this shows that coating bare tapered fiber by PVA increases its sensitivity and resolution as a relative humidity sensor.

Table 4.1: Performance analysis of tapered fiber performance as relative humidity sensor.

Diameter	2.6 μm	2.6 μm PVA	2.6 μm PMMA
Sensitivity (dBm/ %RH)	0.2367	0.3023	0.1936
Linearity (100%)	96.1300	94.19	97.34
Standard deviation (dBm)	0.0223	0.01734	0.1142
Resolution (%RH)	0.0942	0.0574	0.5899
Range (% RH)	35-85	35-85	35-85

Three runs of the experiment were conducted on all the samples. From graphs 4.6, 4.7, and 4.8, the PVA coated sample shows a good repeatability compared to the uncoated one and the PMMA coated. This is due to the fact that the PVA coating is solidified around the tapered region providing additional support to its fragile structure. The fragile nature of uncoated tapered fiber makes more prone to bending and behaving unexpectedly. The PMMA coated showed the largest variations in its three runs, this indicates that using PVA or no coating at all is a better choice.

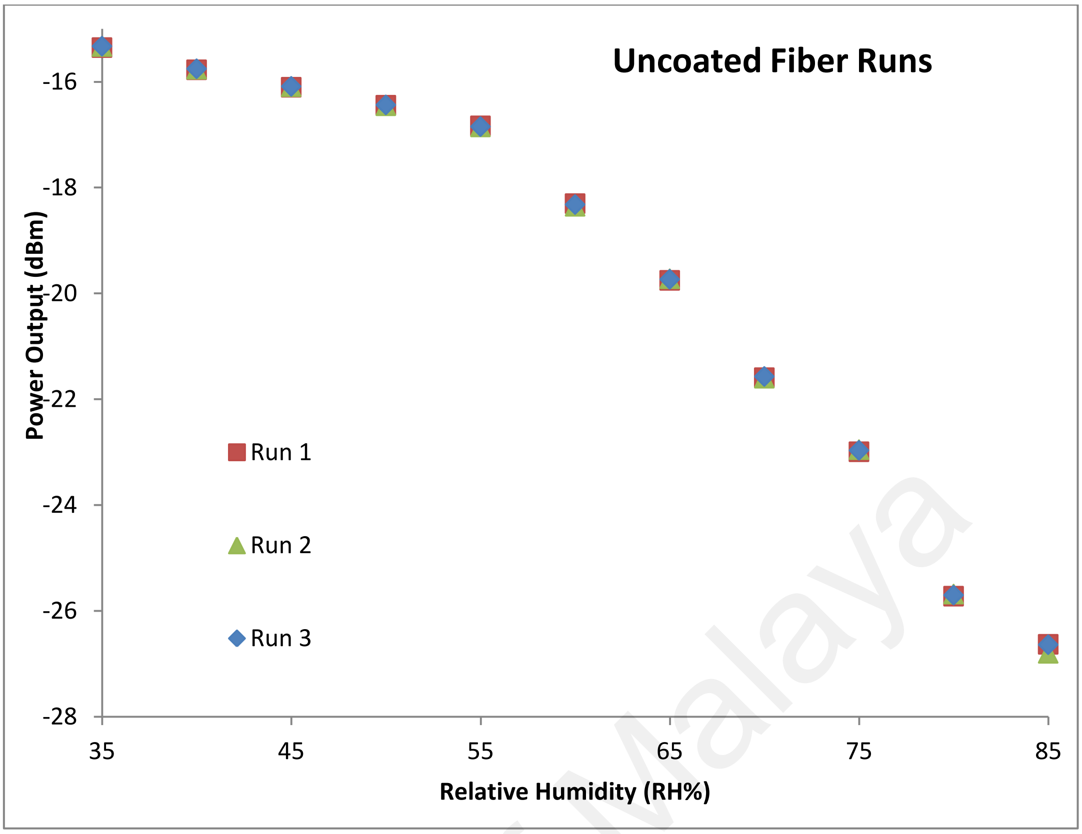

Figure 4.6: The power output of uncoated tapered fiber in three runs.

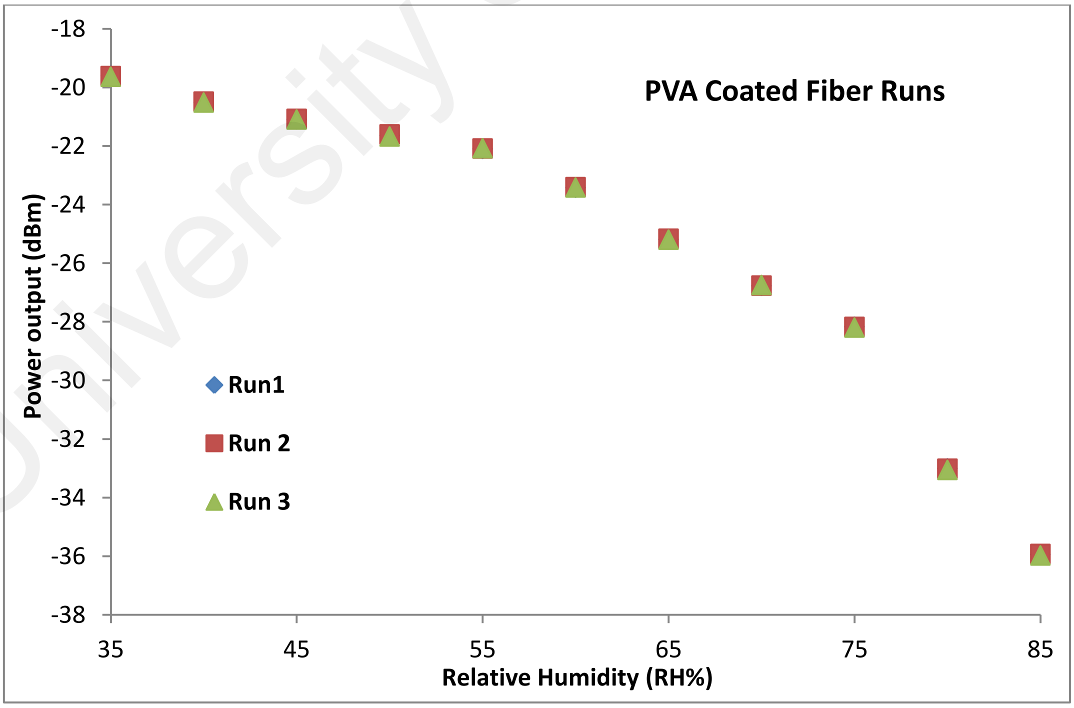

Figure 4.7: The power output of PVA coated tapered fiber in three runs.

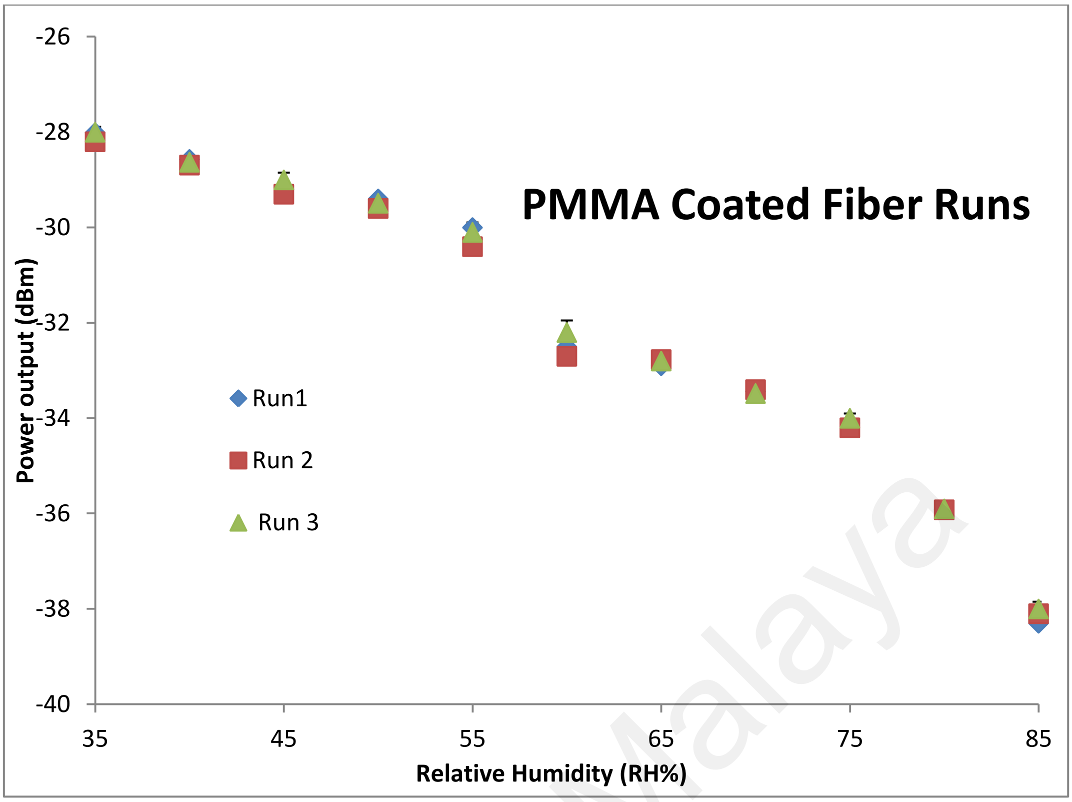

Figure 4.8: The power output of PMMA coated tapered fiber in three runs.

Next, the response and recovery times of the three samples was measured. Each sample was transferred between two locked chambers; one with relative humidity of 85 % RH and the other had 35%RH humidity. The time taken to reach the minimum power output after entering the high humidity chamber is response time. The time to return to the range of larger power output after returning to the 35% RH chamber is recovery time. Both response and recovery times were measured by a stop watch.

Bare fiber had the largest response and recovery times of 7 seconds while the PVA coated sample had a response time of 4 seconds and recovery time of 3 seconds. The PMMA coated sample had the best response time of the three samples with response and recovery times of 2 seconds. This is in line with the results presented in (Peng et al., 2018) , showing that PMMA reacts to changes in relative humidity faster than both PVA and bare fiber do. Despite the fact that is has lower sensitivity, PMMA coating's fast response time makes it a viable choice for relative humidity sensor. The results of these experiments are presented in figures 4.9, 4.10, and 4.11.

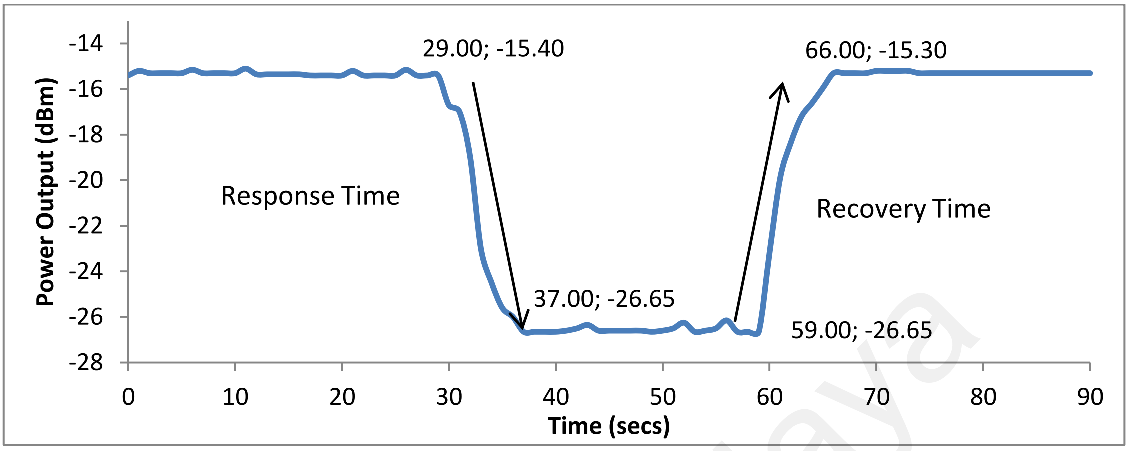

Figure 4.9: The response and recovery time of a 2.6 μm tapered fiber.

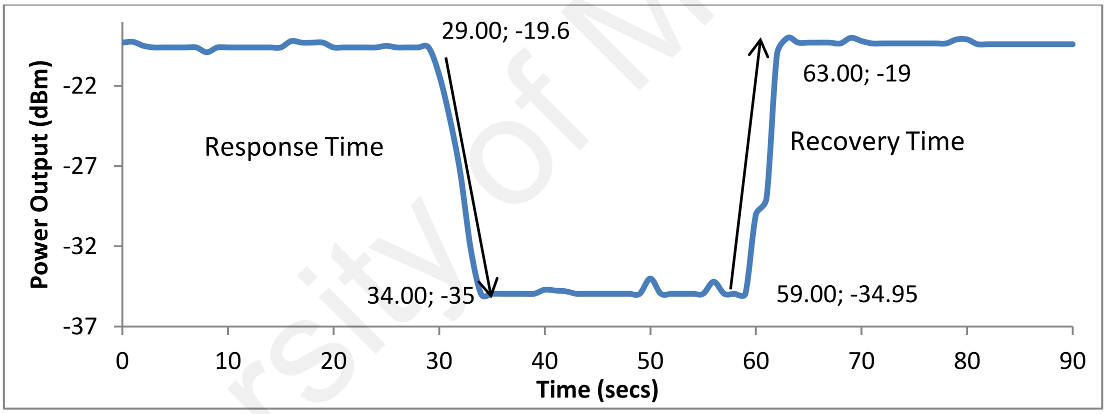

Figure 4. 10: The response and recovery time of a 2.6 μm tapered fiber coated with PVA.

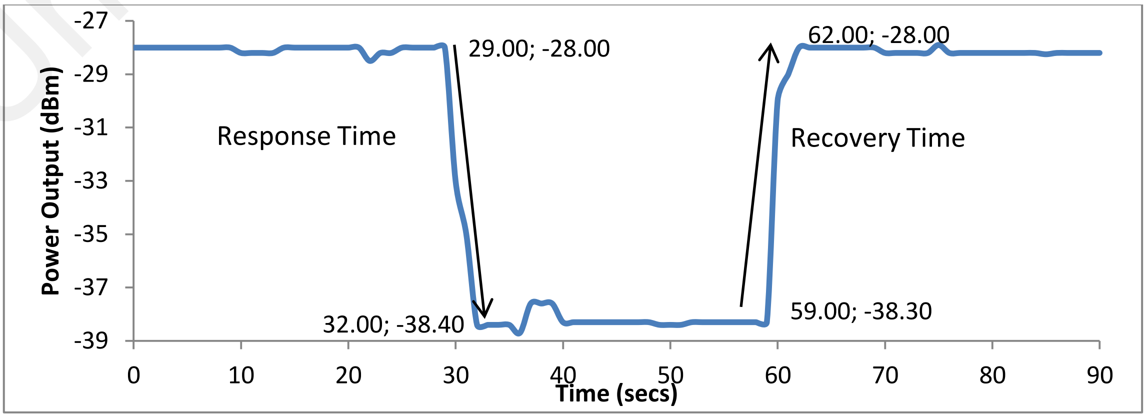

Figure 4.11: The response and recovery time of a 2.6 μm tapered fiber coated with PMMA.

Finally, the stability of our three samples was tested in two time periods; on the day the samples were first prepared and after one week. The power output of each sample was continuously measured for 800 seconds while the samples were in the closed chamber at humidity 60%RH.

On the first day, all samples showed good stability. However, bare fiber and PMMA coated samples were less consistent than the PVA coated sample. After a week, the experiment was repeated. The PVA sample showed good stability. The bare fiber and the PMMA coated sample showed slight variations. The results of this process are presented in figures 4.12 and 4.13. This shows that PVA coated relative humidity sensors are more suitable for constructing relative humidity sensors.

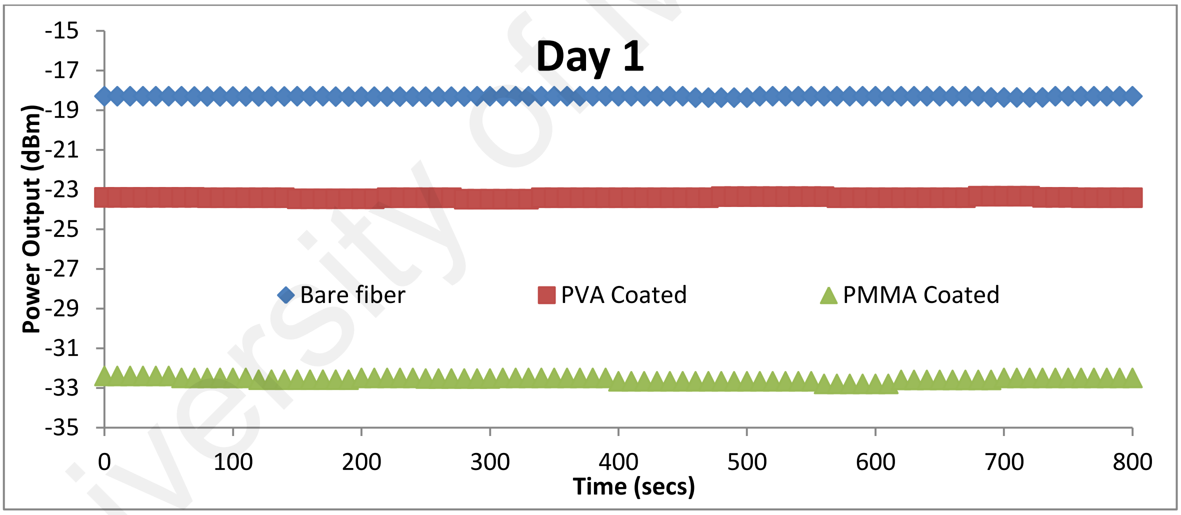

Figure 4.12: Stability of the three samples on the first day.

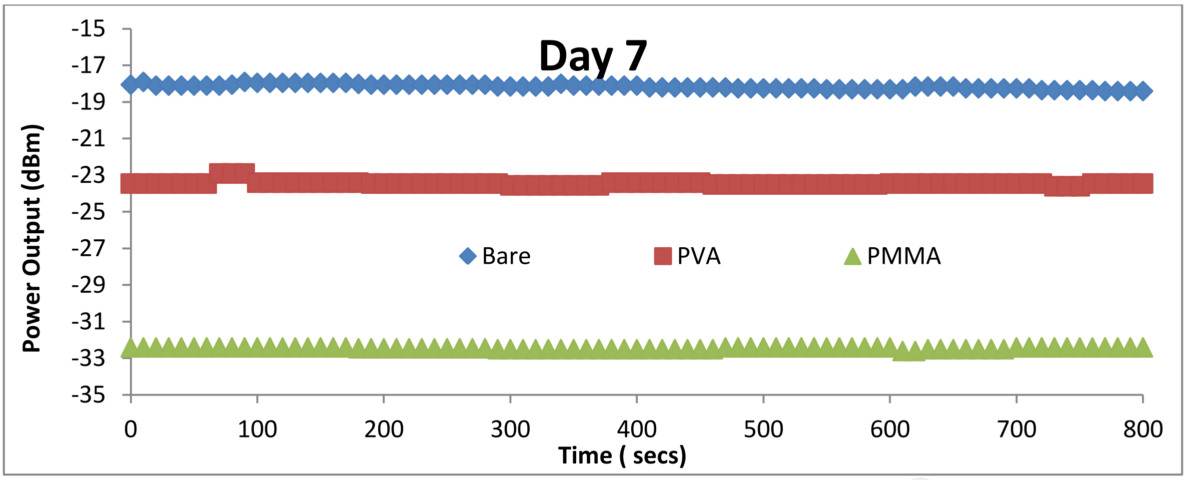

Figure 4.13: Stability of the three samples after seven days.

CHAPTER 5: CONCLUSION

Accurate relative humidity measurements are vital to many industries. Current electronic relative humidity sensors are susceptible to electromagnetic interference and do not work well in volatile environments. Fiber optics based sensors provide a solution to these challenges. Due to their stability, small size, cost, and immunity to electromagnetic interference fiber optics cable can be used to build relative humidity sensors.

In this project, relative humidity sensors were built using tapered fiber optics cable. A single mode fiber was tapered using flame brushing. Afterwards, it was spliced into a patch cord. The power output of the tapered samples was tested by passing light from an ASE source through the tapered sample and into an OPM. The resultant power output was recorded while relative humidity was gradually varied in a locked chamber. The relative humidity in that chamber was recorded using an electronic humidity sensor for reference.

Firstly, we tested the performance of several tapered samples of diameters ranging from 10 to 2 μm. The samples with diameter below 5 μm had a larger sensitivity to changes in relative humidity with the sample of 2.6 μm having the highest sensitivity of 0.2367 db/RH%. This sample size was selected for testing the two coatings for the next stage of this work.

Secondly, several samples of the size 2.6 μm were used for coating. PVA and PMMA were prepared and carefully ripped on these samples. After that, we measured the power output of the coated tapered fiber and compared it to the sensitivity of the bare fiber of the same size.

The results showed that PVA coating increases the sensitivity of the tapered sensors to 0.3023 dB/RH% from the bare fiber's sensitivity of 0.2367 db/RH%. However, the sample coated with PMMA showed the lowest sensitivity of 0.192 db/RH%.

Next, we tested the sensors response time. the three sensors were moved between two chambers with large difference in relative humidity and the time taken for the power output to respond to changes in relative humidity was recorded. The PMMA coated sensor had the fastest response followed by the PVA coated sensor. The uncoated sample was the slowest to respond to changes in relative humidity. Finally, the stability of the samples was tested by continuously measuring the power output of each sensor in constant humidity for 800 seconds. This test was conducted on the first day of constructing the sensors and after a week of that day. On the first day all three sensors performed well. However, the bare fiber showed larger fluctuations with time. After a week, the PVA coated sample was the most stable. The PMMA and bare fiber samples showed more fluctuations after a week. Future work will focus on improving the performance of the PMMA coated sensor in terms of its sensitivity and the PVA coated sensor in terms of its response time.

In conclusion, using tapering provides an effective method to building reliable relative humidity sensors. PVA coating increases the system's sensitivity, while using PMMA gives the system faster response and recovery times.

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