SYNTHESIZE MATERIALS OF OPTICAL FIBRE SENSOR FOR CHEMICAL LIQUID DETECTION

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy (Physics)

Faculty of Science
Universiti Teknologi Malaysia

JULY 2017

To my dearest father, mother, my beloved wife, and my lovely children for their never-ending love, patience and support

ACKNOWLEDGEMENT

First and foremost I would like to thank God, the almighty Allah, for blessing me with sufficient time and health to get my goals. Then, I would like to express the deepest gratitude to my supervisor, Dr. Raja Kamarulzaman Raja Ibrahim, for all the guidance, advice, and support during my PhD study in Universiti Teknologi Malaysia. I have been inspired by his vision and passion to research, his attention and curiosity to details, his dedication to the profession, his intense commitment to his work, and his humble and respectful personality. During this most important period in my research, I thoroughly enjoyed working with him, and what I have learned from him will benefit me for my whole life.

I also would like to express my deepest acknowledgement for the support and help of all my professors, especially to my previous supervisor, Retired Professor Dr. Rosly Abdul Rahman, and my co-supervisors Dr. Norizah Bt Hj Redzuan. I would be my immense pleasure to acknowledge my fellow lab mates. My thanks also go to Mr. Mohd Nasir, Mr. Mohd Sauffie, Mr. Rashdan in the mechanic workshop and the entire Fiber Optic laboratory members (Laser Center, Faculty of Sciences, Universiti Teknologi Malaysia) for their technical assistances.

I would like to express my sincere and wholehearted gratitude to my parents, for their endless love, support, guidance, and inspiration throughout my life. Without their encouragement, I would not be able to achieve everything I have today. They are and always will be my source of strength and courage. A heartfelt gratitude also goes to my brothers, sisters, parents-in-law, and brothers and sisters-in-law for their care and love.

And last, but not least, I would like to give special thanks to my beloved wife, for being extremely supportive during my PhD study. My life would not be so beautiful without her love. Thanks to my lovely children, Mohammed, Sedra, Abdulrahman, for bringing endless happiness into my family.

ABSTRACT

This study aims to improve the detection sensitivity and to reduce temperature effect of the optical fibre chemical sensor (OFCS) system. This system was built for chemical liquid leakage detection using optical time domain reflectometer (OTDR) as an analyzer and unclad multimode optical fibre (MMF) as a sensing part (MMF sensor). A chemical etching technique was used to unclad 1 cm – 3 cm MMF fibre. The OTDR is used as analyzer to record return loss from the OTDR trace. The scale of return loss is determined by the refractive index of chemical sample which is in contact with the MMF sensor. To improve the sensitivity of the OFCS system, the MMF sensor is coated by a thin layer of gold nanoparticles (AuNPs) using sputtering method. The performance of the system is further analyzed to characterize the return loss in relation to the sensor length and the temperature variation. The results show that the sensitivity of the OFCS system using the unclad 3 cm MMF sensor tested with Cargille solutions (refractive indexes 1.440 – 1.474) was found to be 1.825 dB/RI. The best sensitivity of 18.293 dB/RI was achieved when the unclad 3 cm MMF sensor was coated with 14.3 nm AuNPs. The MMF sensor coated with silicon carbide (SiC) was conducted in this study to reduce the influence of the temperature on the sensor response. Analyses on the results reveal that the temperature sensitivity of the OFCS system using MMF sensors coated with SiC and AuNPs was 0.025 $dB/^oC$ and 0.042 $dB/^oC$ respectively when both sensors were tested in toluene. This means the OFCS system coated with SiC is less affected by temperature variation compared to the one coated with AuNPs. However, the sensitivity of OFCS sensor coated with AuNPs to detect the change of the refractive index is much better than the one coated with SiC. Therefore, the optimum performance where sensitivity of the OFCS system to detect the change the refractive index is good and the sensitivity to temperature is minimized was achieved when the MMF sensor is coated with a bilayer coating (AuNPs and SiC). The results reveal that the sensitivity of the OFCS system to temperature with bilayer coating was 0.029 $dB/^oC$. This figure is a little higher than the temperature sensitivity achieved using MMF sensor coated with SiC (0.025 $dB/^oC$). However, it is far better than the MMF sensor coated with AuNPs and yet still shows a good sensitivity to the change in the refractive index.

ABSTRAK

Kajian ini bertujuan untuk meningkatkan kepekaan pengesanan dan mengurangkan kesan suhu kepada sistem penderia kimia fiber optik (OFCS). Sistem ini telah dibina untuk pengesanan kebocoran cecair kimia menggunakan reflektometer domain masa optik (OTDR) sebagai penganalisis dan fiber optik mod berbilang (MMF) tanpa salutan sebagai komponen penderia (penderia MMF). Teknik kakisan kimia telah digunakan bagi membuang salutan MMF sepanjang 1 cm – 3 cm. OTDR digunakan sebagai penganalisis bagi merekodkan isyarat balikan jejak OTDR. Skala isyarat balikan adalah bergantung kepada indeks biasan bahan kimia yang bersentuhan dengan penderia MMF. Bagi meningkatkan kepekaan sistem OFCS, MMF disalutkan dengan lapisan tipis zarah nano emas (AuNPs) menggunakan kaedah percikan. Prestasi penderia dianalisis bagi mencirikan hubungan isyarat balikan dengan panjang penderia MMF dan variasi suhu. Hasil kajian menunjukkan kepekaan OFCS bagi penderia MMF sepanjang 3 cm tanpa salutan ialah 1.825 dB/RI apabila diuji menggunakan larutan Cargille (indeks biasan 1.440 – 1.474). Kepekaan terbaik 18.293 dB/RI diperolehi apabila MMF disalut dengan lapisan tipis zarah nano emas setebal 14.3 nm dan panjang MMF adalah 3 cm. Sistem penderiaan OFCS dengan salutan silikon karbida (SiC) juga telah dilaksanakan bagi mengurangkan kesan suhu ke atas tindak balas penderia. Analisis data menunjukkan kepekaan suhu bagi MMF disalut dengan SiC dan AuNPs masing-masing adalah 0.025 $dB/^oC$ dan 0.042 $dB/^oC$ apabila diuji menggunakan toluene. Ini bermakna sistem penderia OFCS dengan salutan SiC adalah kurang terkesan terhadap variasi suhu berbanding menggunakan salutan AuNPs. Walau bagaimanapun, kepekaan OFCS dengan salutan AuNPs terhadap perubahan indeks biasaan adalah lebih baik berbanding menggunakan salutan SiC. Oleh itu, prestasi optimum iaitu apabila kepekaan OFCS terhadap perubahan indeks biasan adalah memuaskan dan kepekaan terhadap suhu adalah minimum dicapai apabila penderia MMF disalut dengan dwilapisan (AuNPs dan SiC). Keputusan mendedahkan bahawa kepekaan sistem OFCS terhadap suhu menggunakan salutan dwilapisan ialah 0.029 $dB/^oC$. Angka ini adalah lebih tinggi daripada kepekaan suhu dicapai menggunakan penderia MMF disalut dengan SiC (0.025 $dB/^oC$). Walau bagaimanapun, ianya jauh lebih baik berbanding penderia MMF menggunakan salutan AuNPs namun pada masa yang sama menunjukkan kepekaan yang baik terhadap perubahan indeks biasan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

θ_c	-	critical angle
θ_i	-	angle of incidence
θ_r	-	angle of refraction
n	-	refractive index
n_1	-	refractive indices of the fiber core
n_1	-	refractive indix of the fiber core
n_2	-	refractive indix of the fiber clad
n_0	-	refractive indix of the air
Δn	-	Refractive Index Change
NA	-	Numerical Aperture
Δ	-	relative refractive index difference
P_{in}	-	input power
P_{out}	-	output power
P_T	-	transmitted power
L	-	fiber length
α	-	attenuation coefficient
R	-	reflection coefficient
t	-	time of propagating signal
z	-	distance of propagating light
α_s	-	attenuation of Rayleigh scattering
ν_g	-	goup velocity
α_s	-	attenuation of Rayleigh scattering
α	-	total attenuation constant
S	-	the backscatter factor
D	-	pulse width
I_o	-	incident intensity
I	-	transmitted intensity
d_p	-	penetration distance

LIST OF ABBREVIATIONS

OFCS system	-	Optica Fiber Chemical Sensor System
OTDR	-	Optical Time Domain Reflectometer
MMF sensor	-	Multimode Fiber sensor
SMF	-	Single Mode Fiber
MMF	-	Multimode Fiber
RIU	-	Refractive Index
dB	-	decibel
AuNPs	-	Gold nanoparticles
SiC	-	Silicon Carbide
TEOS	-	Tetraethylorthosilicate
CH4	-	Methane
SiH4	-	Silane
N2	-	Nitrogen
Ar	-	Argon
SPR	-	Surface Plasmon Resonance
FBG	-	Fiber Bragg Grating
DBD-NTP	-	Dielectric Barrier Discharge-Non thermal Plasma
EDX	-	Energy Dispersive X-Ray
LSPR	-	Localized Surface Plasmon Resonance
LMR	-	lossy Mode Resonance
dB/UIR	-	Loss Per Unit Refractive Index
KOH	-	Potassium Hydroxid
VHF-PECVD	-	Very High Frequency-Plasma Enhanced Chemical Vapor Deposition

CHAPTER 1

INTRODUCTION

1.1 The Background of Study

Optical fiber sensor is one of the fastest developing technologies today. Fiber sensors have been increased widely, and are used in various sensing applications. Fiber optic sensor has developed to detect the physical parameters, such as strain, temperature, vibration and chemical leakage (Xu *et al.*, 2017). It has numerous advantages compared to the conventional electrical sensors such as, light weight, small size, high-temperature sensitivity, dielectric, immune to electromagnetic interference (Udd, 1995).

Recently, optical fiber chemical sensors (OFCS) system rises the interacts in the detection of different pollutants in the environment, which are leading to toxicity and severe problems of human health. The OFCS system is used to detecte the chemical species (Ho *et al.*, 2001; Bandodkar *et al.*, 2016). The chemical leakage is susceptible to many possible hazards which make conventional electric sensors unfeasible. Thus, Optical fiber sensor can be used to overcome the problem associated with an electrical based sensor.

Optical Fiber chemical sensoes using multimode fiber and single mode fiber (Saini *et al.*, 1995, 2001), or special optical fibers (Morris *et al.*, 1992; Yang and Wang, 2007), are the best possible options. The OFCS can be an extrinsic and intrinsic sensor. However, the extrinsic sensors generally exhibit connection problems. The sensitivity is considered to be one crucial prerequisite for the success the fiber sensor system technology. This is related to the refraction index of the fiber. In addition to the index of the refraction of the fiber, the so-called evanescent field can be improved to reach the high sensitivity of the fiber sensor (Grattan and Sun, 2000).

The OFCS system is required in different fields of application such as, the factories and plants chemical industrial (Partridge *et al.*, 2014; Alwis *et al.*, 2016), landfills, chemical delivery pipelines (Sivathanu, 2003), and fuel station to enable preventive measures and stop the damage from the chemical leakage. Several types of optical fiber sensors have been proposed such as Surface Plasmon Resonance (SPR) (Velazquez-Gonzalez *et al.*, 2017), Fiber Bragg Grating (FBG) (Woyessa *et al.*, 2017) Hetero-core OFS (Iga *et al.*, 2005), tapered optical fiber (Yang *et al.*, 2017), chemically etched OFS (Yun *et al.*, 2012), surface-enhanced Raman spectroscopy (Viets and Hill, 2000), Fabry-Perot interferometric (Pawar *et al.*, 2017) and Optical Time Domain reflectometer (OTDR) (Sumida *et al.*, 2005).

In additional, to fabricate the fiber optical sensor, the simple technique must be the alternative option. Chemical etching technique is an inexpensive way to fabricate the optical fiber sensors. Using this technique, the cladding diameter of the fibre is reduce substantially or entirely removed (Puygranier and Dawson, 2000; Zhang *et al.*, 2010). In this case, the surrounding chemicals become the cladding of the optical fiber. The etching process increases the evanescent wave of the propagating light in the fiber to penetrate into the surrounding chemicals (Zhang *et al.*, 2010).

Currently, fiber sensors have expanded in its exploration lines and possibilities with use of nanocoating deposition techniques (El-Sherif *et al.*, 2007; Ascorbe *et al.*, 2016). Nanostructured thin films and nanocoatings have been applied to the optical fiber sensor system to fabricate new sensors. In this case, the sensing part, i.e., the sensing section preparation of modified cladding involves two major steps: removal of the cladding of fiber, and coated with of new material which interacts with the surrounding chemicals (Khalil *et al.*, 2004; Nguyen *et al.*, 2016). The thin film of the materials that coated the fibre increases the interaction between the propagating light in the optical fibre with the surrounding chemicals. This coating acts as sensitive material, and therefore its composition and fabrication parameters are thoroughly studied in order to improve sensitivity or other desirable sensing values (Garcia-Ruiz *et al.*, 2016; Rithesh Raj *et al.*, 2016; Zhou *et al.*, 2016).

Furthermore, locating the leak chemicals is an important point in the detection process. One of the most important methods used in locating the leak is an Optical Time Domain Reflectometer (OTDR) technique. The OTDR technique can easily determine the location of the chemicals leakage (Juarez and Taylor, 2005; Sumida *et al.*, 2005). The OTDR technique is widely used for the detection location of fault in optical fiber telecommunication system such as, fiber break, bending as well as the splicing points and connectors points along of the fiber. It was employed extensively the sensing system to detect different parameters such as, chemical leakage, temperature, pressure and strain (Potyrailo and Hieftje, 1998; Grattan and Sun, 2000). The major benefit is that the OTDR technique can be used as source and detectors simultaneously, thus it can be connected to one end of the optical fiber. Hence, this technique has become a practical, helpful tool in manufacturing, testing, and installing optical fiber cables.

This research aims to study the sensor response of chemicals leakage by constructing and designing optical fiber chemical sensor. We proposed optic fiber chemical sensor system (OFCS) based on the OTDR as the interrogation system and the Multimode fiber (MMF) as sensor part. The OFCS is constructed to address two main challenges that normally encounter in any optical fiber sensor system. The first one is to increase the sensitivity of the OFCS by etching the cladding of MMF sensor and replace it with a gold layer. Second, to reduce the effects of the temperature in the OFCS response is reduced using silicon carbide layer (SiC).

1.2 Problem Statement

Utilizing detection sensors for chemical liquid leakages that can occur in a fuel station, a tank store or an oil refining is very crucial for safety issues. This is due to many chemicals have hazardous properties (i.e. fire, explosion) and/or health hazards (i.e. toxicity, chemical burns, and dangerous fumes). Thus, many researchers have proposed fiber optic sensors for chemical liquid leakages, which offer many benefits over conventional electrical sensors such as, no current involve, higher resolution and higher accuracy. Moreover, chemicals leakages can be monitored in the systems based optical fiber sensors.

Different techniques can be used to fabricate and design the optical fiber chemicals sensor systems such as Surface Plasmon Resonance (SPR), Fiber Bragg Grating (FBG) and Optical Time Domain Reflectometer (OTDR). SPR and FBG techniques require complex devices and need process sensor for the manufacturing. However, a simple and an accurate measurement technique required for the sensor systems. Furthermore, the sensor systems can be used for long distances to detect the chemical leakages. Therefore, OTDR technique can be used to offer a simple way to detect the leakage of chemicals, as well as it can be employed for long distances monitoring. Also, the OTDR technique becomes most effective when testing long fiber with splices and connectors.

In this work, we used gold nanoparticles (AuNPs) coating, which is well known as the stable material, to solve the problem of low sensitivity of the optical fiber sensor systems. The MMF sensor was coated with a thin layer of the AuNPs to increase the interaction between the sensor section and the surrounding medium. However, due to the low capacity heat of the AuNPs, it found that the system detection coated with the AuNPs considerably influenced by raising the temperature of the sample. Thus, to address the problem of temperature influence, the Silicon Carbide film (SiC) was utilized as an additional layer on the MMF sensor. In addition, the SiC film, which has high capacity heat, was used as a heat sink layer to reduce the temperature effects on the OFCS systems.

1.3 Objectives of the Study

The main objectives of the project are the manufacture of optical fibre chemical sensors , the characterization of their properties, study of the sensor response to the chemicals by deposition of a nanostructured coatings and demonstration of their application as sensors. The goals in the realization of the project are:-

i. To construct and develop the OFCS system based OTDR technique.
ii. To increase the sensitivity of OFCS using AuNPs toward the refractive index of the sample and temperature variation.
iii. To decrease the effects of temperature variation of the sample on the OFCS using SiC as a heat sink.

1.4 Scope of study

The particular focus of this project is limited to the detection and monitoring of chemicals leakage. We seek suitable sensors that can be emplaced and operated with minimal management. In this study, the constructed OFCS system is based on the Multimode fiber (MMF) as sensor section and the OTDR as analyzer. The MMF sensor was fabricated using chemical etching method, which is one of the common and useful method that used in the field of optical sensors to remove the cladding of the silica fiber, as well as it gives good results during the sensor fabricating process.

The current study contributes to addressing two important issues. The first is to increase the sensitivity of the OFCS system using of AuNPs coating. Different thickness of the AuNPs was deposited on the MMF sensor using the sputtering method to optimise the sensitivity of OFCS system. The second one is to decrease the effects of temperature variation on the OFCS system using the SiC. In this study, different experiments were conducted consecutively to deposit the SiC film on the MMF sensor, which can be used to reduce the influence of temperature on the OFCS system. Additionally, different thicknesses of SiC were deposited using two techniques which are Dielectric Barrier Discharge Non-Thermal Plasma system (DBDNTP) and Very High-Frequency Plasma Enhancement Chemical Vapor Deposition (VHF-PECVD). Furthermore, the experiment for etching the SiC film also investigated in this study. Additionally, the potassium hydroxide (KOH) was selected as the solvent solution to remove a small portion from the SiC film. Besides, various etching experiments were carried out at room temperature to produce the cracks on the surface of the SiC film, which allows the sample come through SiC film and then increases the interaction between the MMF sensor and the sample.

1.5 Significance of the research

In recent years, there has been increasing interest in the development of optically based chemical sensors. The optical fiber chemical sensor is a device that can be used to measure the concentration or activity of a chemical species in a sample of interest. Furthermore, the optical fiber sensor can be developed by nanostructure coatings deposition to increase their sensitivity. Additionally, the optical fiber sensors can used to detect the change in the temperature of the chemical liquid.

Many techniques have been used to characterize the sensing part such as Surface Plasmon Resonance (SPR) and Fiber Bragg Grating (FBG). However, these techniques require complex devices and need process sensor for the manufacturing. The Optical Time Domain Reflectometer (OTDR) technique is offer a simple way and accurate measurement which can be used to characterize the sensing system. In this study, the optical fiber sensor based on the OTDR technique has capability to detect the chemical leakage in different fields, such as fuel station, oil pipeline underground, deep wells and tanks storage.

1.6 Summary of Thesis

Chapter 2 provides the historical introduction of the optical fibre technology and introduces optical fibres sensor, with their manufacture techniques. This chapter describes fiber-optic systems in a comprehensive manner. Also, the propagation of light, the fundamental physics and principles of optical fiber geometrical were included. Furthermore, the basic concepts, application, techniques and types of the optical fiber sensors were presented in this chapter. Moreover, the principle of the OTDR technique was also covered in this chapter. Finally, the literature review of optical fiber sensor coated with both of AuNPs and SiC were introduced at the end of this chapter.

In chapter 3, the experimental setups and methods used to fabricate the sensor section are introduced. First, the chemical etching technique to remove the cladding of fiber was conducted. The second part covered the coating of the sensor section with AuNPs and SiC respectively. The experiment setups of the OFCS system for different chemicals, different length of the MMF sensor and different temperature were presented in end of this chapter.

Chapter 4 presents the experimental results and a discussion of the results of this work. The results of four experiments were conducted in this chapter. The first experiment represents the results of the OFCS system without coating. Moreover, the influence of the different refractive index, different length of the MMF sensor and different temperature on the OFCS system without coating were presented in this chapter. The second and third experiments include the results of the OFCS coated with AuNPs and SiC respectively. Furthermore, the final experiment involved the results of the OFCS system coated with bilayer (AuNPs and SiC were introduced at the end of this chapter.

Chapter 5 which is the last chapter of thesis includes the conclusion and the recommendation of this study.

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