UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF FIBER BRAGG GRATING BASED ETHANOL SENSORS USING NANOSTRUCTURED SENSITIVE LAYERS

PUNITHAVATHI THIRUNAVAKKARASU

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By

PUNITHAVATHI THIRUNAVAKKARASU

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

April 2016

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Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Doctor of Philosophy

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April 2016

Chairman : Assoc. Prof. Ahmad Shukri b. Muhammad Noor, PhD
Faculty : Engineering

Optical fiber sensors are widely gaining popularity over the past two decades as the call for highly efficient, miniaturised and versatile sensing devices are rapidly becoming a necessity. This research focuses on designing and analysing a standard single mode Fiber Bragg Grating (FBG) to be used as a sensor for the detection of aqueous ethanol. FBG sensors are conventionally used in the telecommunication wavelength for the measurement of physical parameters such as temperature, strain, pressure and so on. In this research, the FBG sensor is exploited in the ultraviolet and visible (UV VIS) region.

Most optical fiber sensors require some modification to their structure such as removal of the cladding or tapering, in order to enhance the evanescent wave at the interface between the fiber and the surrounding medium. This makes the fiber fragile and difficult to handle. In the UV VIS region, the gratings in the FBGs function as refractors that push the light out to the cladding enabling the interaction of the light signal with the medium surrounding the fiber. This behaviour of the FBG makes it possible to be used without the removal of the cladding making it a very robust and reliable sensing device. The enhancement of the sensing capability is explored using gold layer to boost the evanescent field surrounding the optical fiber Bragg grating. The effect of nanostructured layers of graphene oxide (GO), carbon nanotubes (CNTs) and zinc oxide (ZnO) are investigated towards the performance improvement of the optical sensor. GO, CNTs and ZnO have emerged as leading materials in wide variety of applications including chemical sensors due to its exceptional thermal, optical and mechanical properties. Their nanostructures have huge surface area that enhances the sensor-analyte interaction and thus, improves the sensing performance. These nanostructured materials also react chemically with ethanol molecules, resulting in an increase in the sensitivity of the sensor.

For the first time, a fully cladded FBG optical fiber chemical sensor with a thin gold film and nanostructured sensing layer is reported. The sensor was interrogated in the UV VIS region and both the absorbance response as well as wavelength shift due to

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Surface Plasmon Resonance (SPR) were investigated. The CNT coated FBG sensor demonstrated high sensitivity in absorbance levels up to 0.25/vol% ethanol and with a response and recovery time of approximately 50 and 70 seconds respectively. The GO coated FBG sensor demonstrated high SPR wavelength shift at an average of 1000nm/RIU. As a result of this PhD research project, several novel FBG sensors with nanostructured thin films were developed and investigated towards ethanol sensing.

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Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMBANGUNAN PENDERIA ETANOL MENGGUNAKAN GENTIAN PARUTAN BRAGG BERSALUT LAPISAN PEKA BERSTRUKTUR NANO

Oleh

PUNITHAVATHI THIRUNAVAKKARASU

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Pengerusi : Prof. Madya Ahmad Shukri b. Muhammad Noor, PhD
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Penderia gentian optik semakin popular sejak dua dekad yang lalu memandangkan keperluan yang semakin tinggi bagi peralatan penderia yang tepat, pantas, bersaiz kecil dan serba boleh. Kajian ini memberi tumpuan kepada merekabentuk dan menganalisa gentian parutan Bragg (fiber Bragg grating - FBG) untuk digunakan sebagai penderia bagi mengesan etanol di dalam perantara akueus. Dalam kajian ini, penderia FBG digunakan di rantau panjang gelombang ultraungu dan nampak (UV VIS).

Kebanyakan penderia gentian optik diubahsuai strukturnya, seperti penyingkiran lapisan klad atau di tiruskan untuk meningkatkan penembusan gelombang evanesen pada antaramuka gentian optik dan persekitaran. Ini menjadikan gentian optik rapuh dan sukar untuk dikendali. Di rantau panjang gelombang UV VIS, parutan Bragg berfungsi sebagai pembias yang memantulkan cahaya keluar ke lapisan klad lantas membolehkan interaksi antara isyarat cahaya dan perantaraan di sekeliling gentian optik. Sifat FBG dalam rantau panjang gelombang ultraungu dan nampak inilah yang membolehkannya untuk digunakan tanpa penyingkiran pelapisan klad, dan ianya menjadikan gentian optik ini sangat kukuh dan berkesan. Keupayaan penderiaan ditingkatkan menggunakan lapisan emas untuk menguatkan penembusan gelombang evanesen sekitar FBG. Kesan lapisan berstruktur nano grafin oksida (GO), tiub nano karbon (CNT) dan zink oksida (ZnO) dikaji ke arah peningkatan prestasi penderia gentian optik. GO, CNT dan ZnO telah muncul sebagai bahan peneraju dalam pelbagai jenis aplikasi termasuk penderia kimia kerana sifat-sifat termal, optik dan mekanikal yang luar biasa. Struktur nano tersebut mempunyai luas permukaan yang besar yang meningkatkan interaksi antara permukaan penderia dan analit justeru, meningkatkan prestasi penderiaan. Bahan-bahan berstruktur nano tersebut juga berintreraksi secara kimia dengan molekul etanol dan meningkatkan kepekaan penderia.

Buat kali pertama, kajian menggunakan penderia kimia FBG yang tidak dibuang lapisan salut dan dilapiskan bahan berstruktur nano, terhadap etanol dilaporkan. Penderia ini digunakan di rantau UV VIS dan kedua-dua tindak balas serapan dan juga anjakkan panjang gelombang Surfacr Plasmon resonance (SPR) telah dikaji. Penderia FBG bersalut CNT menunjukkan kepekaan yang tinggi dalam serapan sehingga

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0.25/vol% etanol serta masa tindak balas dan pemulihan kira-kira 50 hingga 70 saat masing-masing. Penderia FBG bersalut GO menunjukkan perubahan panjang gelombang SPR yang tinggi pada purata 1000 nm / RIU. Hasil daripada projek penyelidikan PhD ini, beberapa sensor FBG novel dengan filem-filem nipis bahan berstruktur nano telah dibentukkankan dan disiasat terhadap pengesanan etanol.

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I certify that a Thesis Examination Committee has met on 13 April 2016 to conduct the final examination of Punithavathi a/p M.Thirunavakkarasu on her thesis entitled "Development of Fiber Bragg Grating-Based Ethanol Sensors using Nanostructured Sensitive Layers" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
Au	Gold
BTU	British Terminal Unit
CCD	Charge Coupled Device
CH_3CH_2OH	Ethanol
CNT	Carbon Nanotube
DC	Directional Coupler
EDX	Energy Dispersive X-Ray Spectroscopy
EIA	Environmental Impact Association
FBG	Fiber Bragg Grating
FESEM	Field Emission Scanning Electron Microscopy
GO	Graphene Oxide
H_2O_2	Hydrogen Peroxide
H_2SO_4	Sulphuric Acid
HCl	Hydrochloric Acid
HF	Hydrofluoric Acid
IR	Infrared
$KMnO_4$	Potassium Permanganate
LEL	Lower Explosive Limit
LPG	Long Period Grating
LSPR	Localised Surface Plasmon Resonance
MEMS	Microelectromechanical Systems
MMF	Multimode Fibers
MWCNT	Multi Walled Carbon Nanotube
NIR	Near Infrared
OECD	Organization for Economic Cooperation and Development
OSA	Optical Spectrum Analyser
PCF	Photonic Crystal Fiber
PMMA	Polymethylmethacrylate
POF	Polymer Optical Fiber
RI	Refractive Index
SNR	Signal to Noise Ratio
SMF	Singlemode Fiber
SPP	Surface Plasmon Polariton
SPR	Surface Plasmon Resonance
SWCNT	Single Walled Carbon Nanotube
TIR	Total Internal Reflection
UEL	Upper Explosive Limit
UV	Ultraviolet
VIS	Visible
VOC	Volatile Organic Compound
WDM	Wavelength Division Multiplexing
XRD	X-Ray Diffraction
ZnO	Zinc Oxide

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CHAPTER 1

INTRODUCTION

1.1 Overview

The future of optical fibers as a transmission medium in telecommunication has been firmly established over the past few decades. However it is exciting to note that in recent years, diverse applications of optical fibers are being reported such as in the areas such as illumination [1]–[3], medical diagnostics and treatment [4]–[6] and optical sensing [7]–[9]. Keen interest has been reported in the research and development of physical, chemical and biological optical sensors. A large variety of optical sensing technologies exist and their development is subject to a number of driving factors. For example, in the fields of environmental monitoring [10]–[12], there is a desire to monitor concentrations and compositions of hazardous chemicals in real time. It is useful to be able to perform this monitoring using a device that is safe in a potentially volatile or flammable environment where intrinsically safe technology is a prerequisite. Optical fiber sensors, owing to their capability to be used in such hazardous environments is gaining much popularity. Due to their telecommunications origin, optical fiber sensors are easily integrated into optical networks and telecommunications systems with the added capability of distributed and remote sensing.

Volatile organic compounds (VOCs) are classified as a group of carbon based chemicals that tend to evaporate at room temperature [13][14]. VOCs are widely used in many industrial and academic settings as solvents, cleaning and disinfecting agents and even fuel. Although most VOCs are not acutely toxic, prolonged exposure to them can lead to multiple long term health problems [13] [15]. As the concentrations of these VOCs in the environment are usually low and symptoms take time to develop, there is a necessity to develop a real time sensor capable of monitoring these levels in the environment to prevent a health risk.

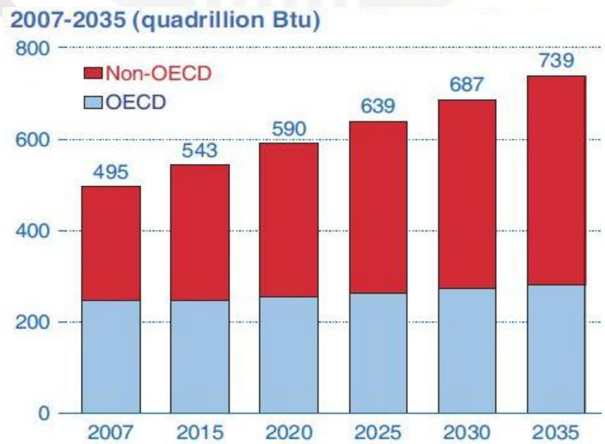

Figure 1.1. World energy consumption projection [16]

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Ethanol is a volatile organic compound that is generally produced through fermentation process of sugars by yeast. It is also knows as Ethyl Alcohol or Ethan-1-ol and has a chemical formula CH3-CH2-OH or simply as C_2H_5OH. The common type of ethanol is found in alcoholic beverages, which can cause intoxication when consumed. It is a volatile, flammable, colourless liquid which is also used as an antiseptic, solvent and is currently gaining popularity and being used as fuel.

Around the world, the demand for renewable and clean energy is constantly increasing. Researchers are looking into many alternative sources of renewable energy to fulfil this demand. The Environmental Impact Association (EIA) recently released its projection, depicted in Figure 1.1, that the world's energy consumption will grow by 56% between 2007 and 2035 which is including Organization for Economic Cooperation and Development (OECD) and Non-OECD nations[16]. Ethanol is a renewable energy produced primarily from agricultural sources. It produces considerably less harmful emissions as it burns much cleaner than [17]. It has become a strong contender to reduce the world's dependence on fossil fuels.

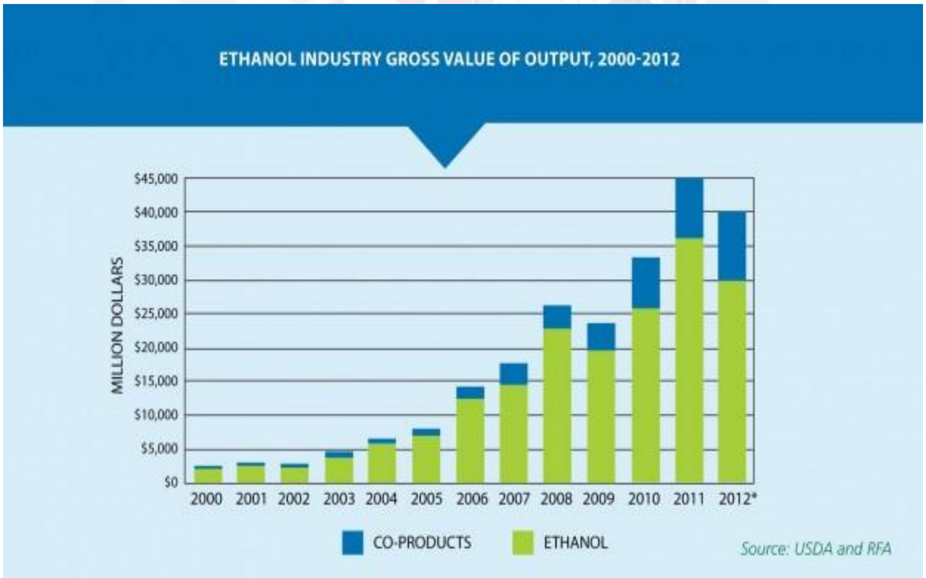

Figure 1.2. Ethanol production from 2000 to 2012 [16]

Over the past decade, ethanol production has risen substantially due to the rise in demand for ethanol blend fuels. It was reported that 13 billion gallons of ethanol was transported, in the United States of America (USA), either by rail or barges, from production facilities in 2010[16]. Ethanol is currently the largest volume hazardous material shipped by rail. Figure 1.2 shows the increase in production volume of ethanol in the USA from 2000 to 2012.

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One of the major concerns is leakage or spills during transportation or storage. Liquid ethanol is flammable and colourless and it is completely miscible in water. Its mass is heavier than air, and the combustible range of ethanol is much wider than gasoline. Ethanol has a Lower Explosive Limit (LEL) to an Upper Explosive Limit (UEL) range of 3.3% to 19% [17]. Pure ethanol has a very low flash point which is 13° C [17]. Although its flammability is affected if mixed with water, however, ethanol remains flammable even if its presence in water is only 20%. At this concentration, it has a flash point of 36°C, at which it is still considered to be a highly volatile and flammable liquid.

Exposure of humans to ethanol during spills or leaks usually involve inhalation, direct skin contact or ingestion, in cases where the ethanol enters water catchment areas (surface water intakes or groundwater). Some of the major impacts attributed to ethanol spills have been to surface water. Fatality of aquatic species have been reported to occur up to several days following ethanol spills, due to the depletion of oxygen in the water. Often, these occurrences happen even up to some distance from the location of the original spill. Once ethanol spills are discharged into a larger bodies of water, response options are very limited. It had been reported that, in the USA, from 2006 to 2010, there were 11 major spills reported, mostly resulting in water contamination [17]. Therefore, continuous monitoring of ethanol levels in water should be performed in order to determine the water quality is approaching anoxic or toxic levels.

Sensors to accurately monitor the concentrations of VOCs in aqueous mediums are very important in order to avoid potential health and environmental problems. Conventional testing for aqueous ethanol include distillation followed by hydrometry / refractometry, gas chromatography, infrared spectroscopy. Most of these methods involve collecting samples and sending them back to a laboratory and results usually take a few days to obtain. Miniaturised optical fiber sensors with real time and in situ sensing capabilities are ideal for this applications. In hazardous or harsh surroundings, especially considering the hazardous nature of the VOCs, optical fiber sensors also have the capability of remote sensing that is ideal for this application.

1.2 Optical Fiber Sensors with Nanostructured Sensing Enhancements

Optical fiber sensors have gained much popularity and market acceptance in recent years due to the number of advantages compared to their electrical counterparts. These advantages include miniaturization, flexibility, immunity to electromagnetic interference and can operate in hostile environments such as high temperature or chemically reactive surroundings[18]. Optical fibers are also passive devices, therefore, do not conduct electricity, making it safe to be used near flammable or explosive materials such as in oil and gas industries[19].

Recent advances in optical sensing with nanostructured material enhancements are creating a huge impact on the direction of future sensor systems. Rapid progress in nanofabrication methods and nanoparticle synthesis are leading to the development of a variety of new nanostructured materials with unique physical and chemical properties.

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Nanostructures are materials that have at least one dimension in nanoscale range, which is less than 100 nm[18]. These material also tend to be particularly suitable for chemical and biological sensing applications due to the fact that many of these processes occur in these scale [20].

When materials are reduced to nanoscale dimensions, they reveal new and unique properties such high surface-to-volume ratio, high heat capacity, mechanical strength and changes in magnetic behaviour [20]. Distinctive changes in optical properties include reflectivity, absorbance and luminescence.

Optical sensors with nanostructured enhancements is a new and interesting path for the development of chemical sensors. Integration of nanostructured material thin films on optical transducing platforms have demonstrated improvements in sensing performance. The optical techniques generally employed to measure response in chemical sensing applications are based on reflectance, absorbance, fluorescence, surface plasmon resonance (SPR) and luminescence changes which are caused by the interaction between the nanostructured films and different chemical molecules[18].

1.3 Problem Statement

A large number of sensors developed for ethanol sensing are electrical based. These sensors are reliable and sensitive, however have several drawbacks, such as large in size, where the sample has to be sent to a lab, while others require the sample to be in a gaseous form such as the sensors used in breathalysers. Electrical based sensors are also not suitable to be deployed in flammable environments such as for ethanol sensing. In situ monitoring of ethanol requires an electrical source close to the sensing platform that would be a safety concern. Therefore the development of a safe, simple and reliable sensor for monitoring ethanol concentrations in water is highly desirable.

Most optical fiber sensors undergo some modification to its structure to bring the light travelling in the core closer to the surrounding medium. This is normally achieved by tapering the sensing region or by cladding removal, either by wet etching method or polishing. These fibers are difficult to fabricate and handle as the fiber tends to be very fragile and breaks easily. Therefore a robust fiber optic sensor, used without altering the circumference of the fiber is highly advantageous in sensing applications where the sensing platform needs to be cleaned and reused multiple times.

1.4 Objectives and Research Questions

The main objective for this research is to develop a nanostructure based optical fiber sensor for ethanol sensing applications. This includes:

1. To characterise uncladded and fully cladded FBG for ethanol sensing in the 1550nm region.
2. To characterize fully cladded FBG in the UV VIS region.

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3. To synthesise and deposit GO, CNTs and ZnO nanostructured materials on FBGs.
4. To investigate and analyse the optical sensing performance of the developed sensors towards ethanol.
5. To propose the sensing mechanism of the optical sensors developed.

In order to achieve these objectives, the following research questions were outlined:

1. What are the characteristics of FBGs that can be manipulated to make it sensitive to the surrounding environment?
2. What sensing layers are suitable to be used in order to obtain a response to ethanol?
3. What are the suitable deposition techniques for gold and nanostructured material deposition on FBGs?
4. Which optical measurement technique will be deployed to perform the investigation and data collection for the optical sensor?
5. How different are the optical sensing performances for the various nanostructured sensing layers used?

Based on the research questions, the investigation was focused on developing the FBG as a chemical sensor and investigation of suitable metal and nanostructured materials that would enhance the performance of the optical sensor towards ethanol sensing. The author developed an FBG assisted chemical sensor with a thin gold (Au) layer coated surrounding the FBG and nanostructured sensing layers of graphene oxide (GO), carbon nanotubes (CNT) and Zinc Oxide (ZnO) was introduced on top of the gold (Au) layer independently for each investigation.

The nanostructured materials were deposited on different gold coated FBGs using dropcasting technique to analyse their sensing performance via spectroscopy method. The nanostructured material characterization combined with the ethanol sensing performance contributes to the fundamental understanding of the sensing mechanism of the optical sensor.

1.5 Thesis Organization

This report will firstly provide the information needed to understand the rest of the thesis. In Chapter 2, some theoretical background on optical fiber sensing systems, ethanol sensors and nanostructured materials will be discussed. A review of the previous work done in this area will also be presented here. This is followed by the sensor fabrication and testing as well as nanostructured material synthesis and deposition in Chapter 3.

Chapter 4 presents the characterization results of the nanostructures deposited on FBG. Chapter 5 presents the experimental results of the sensing performance of FBG sensor,

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towards ethanol concentrations, in the 1550 nm and Ultraviolet Visible (UV VIS) region. Finally, in Chapter 6, conclusions will be drawn and the contributions of the thesis to the related research area will be discussed along with recommendations for future works.

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