



UNIVERSITI PUTRA MALAYSIA

***DESIGN AND FABRICATION OF FIBER-BASED SURFACE PLASMON
RESONANCE SENSORS***

YUSSER A. TAQI AL-QAZWINI

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RESONANCE SENSORS**

By

YUSSER A. TAQI AL-QAZWINI

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

June 2016

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DEDICATION

I humbly and lovingly dedicate this thesis to my sister, my mother, my late father who is in heaven, and my beloved country, Iraq.



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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

DESIGN AND FABRICATION OF FIBER-BASED SURFACE PLASMON RESONANCE SENSORS

By

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June 2016

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Surface plasmon resonance (SPR) sensors based on optical fibers, with their high level of miniaturization, high sensitivity to variations in the refractive index (RI) of the surrounding medium, and sensing capability in hazardous areas, have attracted a great attention for the past two decades as one of the most advanced real-time and label-free detection technology. The performance of these devices in terms of their sensitivity and detection accuracy is of great importance and it is strongly influenced by the design parameters such as fiber material and geometry, and the properties of the metal layer and sensing medium. This thesis presents numerical simulations to understand the impact of these parameters on the performance of a basic fiber-based SPR sensor implementing a simple waveguide layered structure. The thesis also reports the fabrication of two cost-effective fiber-based SPR sensors. The developed sensors are tested for RI sensing of aqueous media.

A simple planar layered structure is numerically simulated to provide design specifications for fiber-based SPR sensors. The numerical simulations help understand the influence of the properties of the involved layers like metal layer and residual cladding thickness, sensing length, and RI of the sensing medium on the response of SPR sensors.

An SPR sensor based on a tapered multimode fiber is fabricated and experimentally characterized for its performance. The sensor is made by tapering a standard multimode fiber using an automated tapering machine followed by coating the entire taper with gold without masking. The impact of waist-diameter, waist and transition lengths on the performance of the sensor is investigated. A simple, high-performance, yet robust sensor with a spectral width of 140-220 nm and a sensitivity of ~1600-2000 nm/RIU is realized when the waist diameter is set to 25-45 μm with a total taper length of 3-5 mm and 55-nm gold thickness.

A second configuration based on plastic optical fiber is fabricated and analyzed. This fiber brings additional advantages to SPR sensors because of its large numerical aperture, excellent flexibility, and easy manipulation. The implemented chemical etching process with concentrated Acetone preserves the fiber's circular geometry which simplifies the fabrication process and reduces the associated costs compared to side-polished ones. Same setup and sensing solutions are used while the influence of the gold thickness, fiber residual thickness, and sensing length is investigated. A low-cost, sensitive, and robust SPR sensor with an average sensitivity of 1600 nm/RIU and spectral width of 154 nm is realized when a plastic optical fiber is etched from the middle up to 964- μm with 10-mm sensing length and 55-nm gold thickness.



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

REKA BENTUK DAN FABRIKASI PENDERIA PERMUKAAN RESONANS PLASMON BERASASKAN GENTIAN

Oleh

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Penderia permukaan resonans plasmon (SPR) berasaskan gentian optik, dengan tahap pengecilan saiz yang besar, kepekaan yang tinggi kepada perubahan indeks biasan (RI) dalam medium, dan keupayaan peranti di kawasan berbahaya, telah menarik perhatian tinggi sejak dua dekad yang lalu sebagai salah satu teknologi pengesanan tanpa label yang termaju. Prestasi peranti ini dari segi kepekaan dan ketepatan pengesanan adalah amat penting dan ia dipengaruhi oleh parameter-parameter reka bentuk seperti bahan gentian dan geometrinya dan sifat-sifat lapisan logam dan medium penderiaan. Tesis ini membentangkan simulasi berangka untuk memahami kesan parameter ini kepada prestasi yang berasaskan penderia gentian SPR asas menggunakan teknik pandu gelombang struktur berlapis yang mudah. Tesis ini juga melaporkan pembuatan dua penderia SPR berasaskan gentian yang kos efektif. Penderia dibangunkan dan diuji untuk penderiaan RI bagi medium cecair.

Struktur satah berlapis mudah disimulasi berangka untuk menyediakan spesifikasi reka bentuk untuk penderia SPR berasaskan gentian. Simulasi berangka ini membantu memahami pengaruh sifat-sifat lapisan terlibat seperti lapisan logam dan sisa ketebalan klad, panjang penderia, dan RI medium penderiaan pada sambutan penderia SPR.

Penderia SPR berdasarkan gentian pelbagai-mod dan diujikaji bagi menentukan ciri-ciri prestasinya. Penderia direka oleh tirus gentian mod standard di fabrikasi menggunakan mesin penirus automatik diikuti oleh lapisan keseluruhan tirus dengan emas tanpa pelekat. Kesan diameter-pinggang, pinggang dan peralihan panjang kepada prestasi penderia itu dikaji. Penderia yang mudah, berprestasi tinggi, dan teguh dengan kepekaan $\sim 1600\text{-}2000$ nm/RIU dan lebar spektrum 140-220 nm berjaya direalisasikan apabila diameter pinggang 25-45 mikron dengan jumlah panjang tirus daripada 3-5 mm dan ketebalan emas 55-nm.

Konfigurasi kedua penderia berdasarkan gentian optik plastik direka bentuk dan dianalisis. Gentian ini mempunyai kelebihan tambahan kepada gentian SPR kerana apertur yang besar, fleksibiliti yang cemerlang, dan mudah untuk di manipulasikan. Proses punaran dilakukan bagi mengekalkan geometri bulat gentian ini yang memudahkan proses fabrikasi dan mengurangkan kos yang berkaitan berbanding dengan sisi-gilap. Persediaan sama dan larutan penderia bagi mengkaji pengaruh ketebalan emas, sisa ketebalan gentian, dan panjang penderia. Penderia SPR yang direalisasikan, sensitif, berkost rendah dan teguh dengan purata sensitiviti 1600 nm/RIU dan lebar spektrum 154 nm direalisasikan apabila gentian optik plastik terukir dari tengah sehingga 964-mikron dengan 10 mm panjang penderia dan tebal emas 55-nm.



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I certify that a Thesis Examination Committee has met on 20 June 2016 to conduct the final examination of Yusser A. Taqi Al-Qazwini on her thesis entitled "Design and Fabrication of Fiber-Based Surface Plasmon Resonance Sensors" 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

SPR	Surface plasmon resonance
RI	Refractive index
RIU	Refractive index unit
SMF	Single-mode fiber
MMF	Multimode fiber
POF	Plastic optical fiber
SP	Surface plasmon
SPW	Surface plasmon wave
TM	Transverse magnetic
TIR	Total internal reflection
EW	Evanescent wave
ATR	Attenuated total reflection
SNR	Signal-to-noise ratio
FWHM	Full width at half maximum
vis-NIR	Visible-near infrared
PCF	Photonic crystal fiber
LPG	Long period grating
FBG	Fiber Bragg grating
NP	Nanoparticles
LSPR	Localized surface plasmon resonance
UV-Vis	Ultraviolet-visible
MIP	Molecularly imprinted polymer
MEP	Mode expansion and propagation
FDTD	Finite-difference time-domain
PML	Perfectly matched layer
HL	Halogen
SEM	Scanning electron microscope
PMMA	poly(methyl methacrylate)
SMA	SubMiniature version A
FC	Ferrule connector

CHAPTER 1

INTRODUCTION

1.1 Overview

There is a continuing need for the development of simple and efficient methods for real-time sensing in the fields of chemistry, environmental, and biological studies, etc. To meet this ever-increasing demand, a wide-range of real-time and label-free approaches have been developed based on ellipsometry, grating couplers, interferometers, surface plasmon resonance, etc [1]. Among the aforesaid sensing technologies, surface plasmon resonance (SPR) has attracted much attention for the past two decades because of being label-free, non-destructive and highly sensitive detection method. Optical sensors based on SPR technique are very sensitive to variations in the refractive index (RI) of the sensing medium which made them highly efficient for various sensing applications.

Optical SPR sensors have evolved from the basic SPR configuration based on Kretschmann configuration which comprises basic optical components such as a glass prism, a thin metal film, and the sample layer or surrounding medium [2-4]. However, conventional prism-based SPR sensors suffer from some drawbacks such as bulky size with several mechanical and optical moving elements leading to implementation difficulties for remote sensing applications. Thus, to overcome these drawbacks, optical fibers have been deployed as an alternative to the glass prism.

The introduction of fiber-based SPR sensors as an optical sensor in the early 1990s has gained considerable attention thanks to their high level of miniaturization and sensing capability in inaccessible locations. In addition, they are immune to electromagnetic interference and can be used in hazardous areas [5-7]. Since then, fiber-based SPR sensors have been adopted in various applications and huge effort has been made to enhance their performance [8-12].

The remaining sections in this chapter address the motivation, objectives, and organization of this thesis.

1.2 Problem Statements

In fiber-based SPR sensing devices, the sensing element is typically made of a metal-coated optical fiber. The performance of SPR devices in terms of their sensitivity and detection accuracy is strongly influenced by the design parameters such as fiber material, fiber geometry, and the properties of the metal layer and sensing medium. Therefore, this thesis explores the impact of these parameters on the performance of SPR-based RI sensors starting with numerical simulations of a simple planar

waveguide structure, followed by laboratory experiments on different structures of fiber-based SPR sensors.

A typical fiber-based SPR sensor is normally made from silica fiber, either single-mode (SMF) or multi-mode (MMF) fiber. The design of SPR sensors based on SMF needs very high accuracy due to the small amount of power accommodated by the small core of single-mode fibers [13, 14]. In addition, the polarization of light has to be precisely controlled to reduce instability in the sensor output which requires the use of extra components and eventually raises the system complexity and cost [15]. One more downside of SPR sensors made with SMF is that they are very fragile complicating the sensor fabrication [16]. Multimode fibers, on the other hand, offer a much wider range of measurable refractive index despite suffering from low stability due to the intermodal coupling and modal noise. Apart from that, MMF is less fragile and the sensor system does not need a precise control of the polarization state which offers simpler and relatively lower cost option in the design of SPR sensors.

In order to increase the chances of SPR to occur in an optical fiber, an evanescent field is necessary to generate surface plasmon wave at the interface of a metal layer and the surrounding medium. For that purpose, several structures of fiber-based SPR sensors have been proposed and developed such as partial removal of fiber cladding [17], side-polishing [18, 19], and tapering [20-22]. SPR sensors based on tapered optical fibers, particularly in the case of MMF, feature some advantages such as compactness, simplicity, and high sensitivity compared to those in which the cladding is removed or polished [17, 23]. There have been a large number of studies [24, 25] concerning tapered fiber-based SPR sensors but they did not provide sufficient information on the impact of the taper length considering both the taper-waist length and transition lengths. Furthermore, most of these studies involved some masking techniques to cover certain regions of the tapered fiber making the sensor fabrication more sophisticated.

Hence, this thesis experimentally demonstrates the fabrication of an SPR sensor based on a tapered MMF. The influence of design parameters including the waist diameter, taper waist-length and transition lengths on the sensor performance is thoroughly studied. The gold coating process did not involve masking which offers a simpler approach towards the sensor fabrication.

For low-cost applications, plastic optical fiber (POF) offers a good alternative compared to standard silica fibers such as SMF and MMF. SPR sensors based on POF have a number of advantages including design simplicity, low fabrication cost, and flexibility [26-28]. Most of the reported SPR sensors based on POF are made by side-polishing the polymer cladding which damages the circular symmetry of the optical fiber with consequent polarization dependent losses [29]. In these sensors, the field interaction strength is reduced by the circular asymmetry. In addition, splicing side-polished fibers is not an easy thing resulting in alignment difficulties and consequently additional losses. Besides, the process of side-polishing is mechanically complex [30, 31].

Therefore, this thesis also incorporates the fabrication of a low-cost SPR sensor based on an etched POF in which a cladding reduction approach is implemented using chemical etching with a precise control of the immersion time to control the fiber diameter while preserving the cylindrical geometry of the optical fiber. In addition, the impact of design parameters including gold thickness, amount of residual cladding, and sensing length is also investigated.

1.3 Research Objectives

The objectives of this thesis are:

- To use numerical simulation to study the basic performance characteristics of fiber-based SPR sensors using a simple planar waveguide structure.
- To fabricate an SPR sensor based on tapered MMF without the use of masking.
- To fabricate an SPR sensor based on an etched POF.
- To utilize the developed sensors for RI sensing and analyze their performance in terms of sensitivity and detection accuracy.

1.4 Outline of the Thesis

This thesis consists of six chapters organized as follows:

Chapter 1: INTRODUCTION begins with a brief background in the context of fiber-based SPR sensors. After analyzing the research motivation, objectives of this work are proposed, followed by the structure of the thesis which outlines the whole work carried out.

Chapter 2: LITERATURE REVIEW reviews the SPR theory and relevant literature on fiber-based SPR sensors. First, the chapter introduces the basics of SPR theory and its sensing principles, followed by an introduction to the prism coupling as the base of conventional SPR sensing. Next, the performance characteristics of SPR sensors are reviewed with the right choice of metals and a study of the properties of the complex propagation constant of surface plasmons. The third section provides an overview of fiber-based SPR sensors and the various available structures are reviewed. Finally, the recent advancements in the field of fiber-based SPR sensors are outlined.

Chapter 3: NUMERICAL INVESTIGATION OF A BASIC STRUCTURE OF FIBER-BASED SPR SENSOR presents a numerical investigation of the impact of main design parameters on the performance of a basic structure of fiber-based SPR sensor using a simple layered structure before going into real experimental work and more complicated structures in the subsequent chapters. The chapter serves as a starting point to understand how the performance of SPR sensors is influenced by the

properties of the involved layers including the gold layer thickness, residual cladding thickness, sensing length, and refractive index of the surrounding environment. In the second section, a brief introduction to the finite-different time-domain method is presented. The third section describes the implemented numerical model based on a simple planar waveguide structure followed by the theory related to the calculation of the normalized transmitted power. Finally, the results of the numerical simulations concerning the refractive index sensitivity and detection accuracy of the simulated SPR sensor are compared and discussed.

Chapter 4: SPR SENSOR BASED ON TAPERED MULTIMODE FIBER introduces the details of the fabrication of an SPR sensor based on tapered MMF, including the tapering process of multimode optical fibers with different tapering parameters, and coating of thin gold films on the tapered region without the use of masking. The experimental setup which comprises of simple components is described. In addition, a study of the refractive index sensitivity and detection accuracy of the fabricated SPR sensor is carried out and the results are discussed.

Chapter 5: SPR SENSOR BASED ON ETCHED PLASTIC OPTICAL FIBER reports the fabrication of an SPR sensor based on etched POF. The detailed procedure with regard to the etching technique is described, and a relationship between the fiber thickness and immersion time is presented. The details of the fabrication process, including the fiber etching and gold coating on the etched region are also introduced. Furthermore, the performance of the fabricated sensor with different design parameters is examined and the results are discussed.

Chapter 6: CONCLUSIONS AND FUTURE WORKS concludes the whole work carried out with the research findings of this thesis, summarizing the achievements made against the objectives identified in Chapter 1. This chapter also describes how the present work can be extended through future work to broaden the scope of the research.

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