LOCALIZED SURFACE PLASMON RESONANCE SENSOR BASED ON GOLD-COATED TAPERED OPTICAL FIBER FOR REFRACTIVE INDEX MEASUREMENT

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DEDICATION

This thesis is dedicated to my beloved parents, family and fiancé, E

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ABSTRACT

The tapered optical fiber (TOF) sensor based on localized surface plasmon resonance (LSPR) was experimentally demonstrated for determination of surrounding liquid refractive index (RI) changes. The reduction of tapered waist diameter was done by flame-brushing method which results in the diameter range of 6 µm to 18 µm. The sensing mechanism is based on the transmission shift in the monitored output spectra. Key parameter optimization of the TOF sensor has been investigated particularly on the coating time of gold nanoparticles (AuNPs) onto fiber substrate. Functionalization of AuNPs by self-assemble monolayer was carried out through dip coating technique. Five different coating periods of 24, 48, 72, 96 and 120 hours have been performed on the TOF and characterized by using field emission scanning electron microscopy (FESEM). Histogram analysis was extracted from the FESEM images in order to determine the average particle’s size while the coating thickness were measured based on the cross-section image analysis. The highest sensitivity of the sensor was recorded at 18.49314 nm/RI corresponding to 48 hours of coating time. The optimum thickness of coating layer was calculated around 53.11 nm along the average particle’s size of 15 nm. Energy dispersive X-ray (EDX) characterization shows 22.4% of gold element on deposited fiber surface which proved the existence of the gold on the tapered structure. The optimized sensor was further examined for its overall performance in terms of stability, reversibility and repeatability over RI range from 1.3324 to 1.4254. Stability test of the TOF sensor suggests that the output signals are stable at low and medium of RI but a little variation on highest RI was recorded around ±0.00594 a.u. For the reversibility check, the TOF sensor acquired good response with little variation of ±0.008 a.u. between ascending and descending measurement order.

ABSTRAK

Sensor gentian optik tirus (TOF) berasaskan resonans plasmon permukaan setempat (LSPR) telah ditunjukkan secara eksperimen untuk menentukan perubahan indeks biasan (RI) cecair sekitar. Pengurangan diameter pinggang tirus dilakukan dengan kaedah penyemburan api yang menghasilkan julat diameter dari 6 μm hingga 18 μm. Mekanisme pengesanan adalah berdasarkan peralihan transmisi pada spektrum output yang dipantau. Pengoptimuman parameter utama sensor TOF telah disiasat terutamanya pada masa salutan nanopartikel emas (AuNPs) ke substrat gentian. Fungsionalisasi AuNPs oleh pemasangan-sendiri lapisan tunggal dilakukan melalui teknik salutan celup. Tempoh salutan yang berlainan iaitu 24, 48, 72, 96 dan 120 jam telah dilakukan pada TOF dan dicirikan dengan menggunakan mikroskop elektron pengimbasan pelepasan bidang (FESEM). Analisis histogram telah diekstrak daripada imej FESEM untuk menentukan saiz purata zarah manakala ketebalan lapisan adalah diukur berdasarkan analisis dari imej keratan rentas. Sensitiviti sensor yang tertinggi dicatatkan pada 18.49314 nm / RI bersamaan dengan 48 jam masa salutan. Oleh itu, ketebalan optimum salutan lapisan adalah dikira sekitar 53.11 nm dengan saiz zarah purata sekitar 15 nm. Pencirian penyebaran tenaga sinar-X (EDX) menunjukkan 22.4% elemen emas terdapat pada permukaan fiber sekaligus membuktikan kewujudan emas pada struktur tirus. Sensor yang dioptimumkan selanjutnya diperiksa untuk mengukur prestasi keseluruhan dari segi kestabilan, kebolehulangan dan kebolehulangannya untuk RI diantara 1.3324 hingga 1.4254. Ujian kestabilan sensor TOF menunjukkan bahawa isyarat keluar stabil pada tahap rendah dan median RI tetapi sedikit variasi terkesan pada RI tertinggi yang dicatatkan sekitar ± 0.00594 a.u. Untuk pemeriksaan kebolehulangan, sensor TOF memperoleh tindak balas yang baik dengan sedikit variasi ± 0.008 a.u. dicatat pada ukuran berkadar menaik dan menurun.

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LIST OF ABBREVIATIONS

FBG	-	Fiber Bragg grating
AuNPs	-	Gold nanoparticles
DCA	-	Dicarboxy acetone
DIW	-	Deionized water
EDX	-	Energy Dispersive X-Ray
EG	-	Ethylene glycol
FESEM	-	Field Emission Scanning Electron Microscopy
$HAuCl_4$	-	Hydrocloroauric acid
HR-TEM	-	High Resolution-Transmission Electron Microscopy
LSPR	-	Localized surface plasmon resonance
MMF	-	Multimode fiber
MPTMS	-	Mercaptopropyl trimethoxysilane
NaCt	-	Trisodium citrate
NaOH	-	Sodium hydroxide
NPs	-	Nanoparticles
OSA	-	Optical Spectrum Analyzer
PCF	-	Photonic crystal fiber
PSA	-	Particle Size Analyser
RI	-	Refractive index
SA	-	Self-assembly
SAM	-	Self-assemble monolayer
SMF	-	Single mode fiber
SPR	-	Surface plasmon resonance
TIR	-	Total internal reflection
TM	-	Transverse magnetic
TOF	-	Tapered optical fiber
UV-Vis	-	Ultraviolet visible spectroscopy

LIST OF SYMBOLS

i	-	Incidence angle
i_c	-	Critical angle
θ_r	-	Reflectance angle
θ_t	-	Transmission angle
E_v	-	Evanescent wave intensity
E	-	Output light intensity
E_o	-	Initial intensity of light
x	-	Amount of time passes
d_p	-	Penetration depth
λ	-	Wavelength
π	-	Phi
n_{co}	-	Refractive index of core
n_{cl}	-	Refractive index of cladding
I	-	Input of light
I_o	-	Output of light
r	-	Reduction factor in attenuation
α	-	Absorption coefficient
l	-	Taper length
C	-	Concentration of analyte

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Optical fibers have been a subject of intense study in many areas ranging from modern telecommunications to material sensing owing to their unique characteristics including miniature size, large bandwidth, low attenuation and immunity towards electromagnetic signal (1). Rapid development in the fabrication techniques is responsible for emergence of many types of optical fibers with different functions including fiber Bragg grating (FBG), tapered optical fiber (TOF), tapered-FGB and hetero-core fibers (2–5).At the moment, an intrinsic sensor based on tapered optical fiber is getting more attention due to its superior advantages. This type of sensor possesses relatively large performance efficiency based on the absorption mechanism of evanescent wave. In the recent years, TOF have been employed for the sensing of hydrogen gas, salinity, water pollutant and refractive index measurement (6–9).

Refractive index (RI) sensing measurements are very important because it is considered as one of the fundamental property of any material that vary based on temperature, pressure and concentration (10). In practice, conventional bulk refractometer is commonly employed for edible food product, industrial production as well as medical purposes. However, the conventional device is considered as non-convenient for in-situ measurements due to its limitation in terms of weight and size. Therefore, TOF approaches are better alternative due to its compactness, high resolution, light weight and multiplexing possibilities. Among many, TOF sensor based on localized surface plasmon resonance (LSPR) shows promising correlation in refractive index sensing.

Surface plasmon resonance (SPR) is commonly applied in various applications. There are several differences between SPR and LSPR in terms of its generated resonance, peak absorbance and sensitivity response. LSPR are oscillations of charge density that confined in metallic nanoparticles (NPs). Any changes of refractive index at surrounding medium of NPs will change the resonant frequency that can be observed in intensity or wavelength of LSPR absorption band (11). Fiber sensor utilizing LSPR offer a variety of advantages including accessibility to difficult places, robustness, simplicity and versatility (12,13). The LSPR application on TOF is proposed in order to enhance the sensitivity of RI sensor (14).

Gold is one of noble metal that commonly used in LSPR phenomena. The plasmon oscillation of free electrons for gold nanoparticles (AuNPs), provides significant enhancement to the scattering and absorption of EM signals within the range of visible region as the results of resonance between the plasmon frequencies. This phenomenon yields an intense colors spectrum and resulted in unique optical properties (15). Sharp SPR absorption peak at the visible region which around 520 nm plays an important role to determine novel physical and chemical properties. Turkevich method is the one of simplest technique in order to synthesis AuNPs which been discovered in 1951 (16)and promising good result. By implement gold nanolayer on TOF, the performance of the sensing activity will be obtained based on intensity or wavelength shifting based on LSPR absorption peak.

1.2 Problem Statement

Conventional optical fibers allow limited access to the evanescent field and shows inefficient light-environment interactions that make it less sensitive in micro-scale sensing applications. Thus, a tapered fiber LSPR sensor is proposed. The significance interrogation of the transmission intensity change due to the evanescent field absorption of immobilized AuNPs on the tapered fiber surface is the key factor for this study. The development of such TOF system provides a more efficient and promising solution to meet the challenge. However, the overall performances of LSPR based tapered fiber sensor need to be carefully studied. Sensitivity, stability, repeatability and reversibility of the proposed sensor are yet to be optimized.

1.3 Research Objectives

The main objective of this study is to develop TOF sensor for detection of RI changes. In order to achieve this goal, specific objectives are listed as:

(1) To fabricate and characterize the uniformed tapering region of silica optical fiber by using flame-brush technique
(2) To characterize and optimize the sensing layer prior to the deposition of AuNPs on the tapered region
(3) To determine LSPR response and overall sensitivity enhancement of the tapered optical fiber performance towards controlled surrounding media.

1.4 Research Scope

This study intends to produce a high sensitivity TOF for detection of liquid media RI changes based on LSPR. Single mode fiber (SMF) and multi-mode fiber (MMF) with core/cladding diameter of 10/125 and 62.5/125 μm are used, respectively. Tapered waist are fabricated in ranges of 6 μm up to 18 μm by using flame-brush technique incorporating oxy-butane torch (burning max at 1800 ^{o}C) as heating element. Ethylene glycol (EG) used as controlled samples with varies RI in the range of 1.3324 to 1.4254. Gold particles in nano-size scale are synthesized by established Turkevich method. Gold nanolayer coating is deposited on the tapered structure by using self-assemble monolayer (SAM) method based on dip coating technique. Five period coating time are varies for 24, 48, 72, 96 and 120 hours in order to determine the optimized coating time based on their sensitivity which obtained from shifting measurement. Characterization equipment implies are Ultraviolet Visible spectroscopy (UV-Vis), Particle Size Analyser (PSA), Field Emission Scanning Electron Microscopy (FESEM) with Energy Dispersive X-ray spectroscopy (EDX) and High-Resolution Transmission Electron Microscopy (HRTEM).

A tungsten-halogen lamp (Ocean Optics HL2000) with wavelength range of 360 to 2400 nm is employed to transmit the light into the TOF. A spectrometer model

C175 Thorlabs with spectral range of 499–1100 nm is used as the light detector to measure the output light intensity of the sensor. The transmission signal are collected and analysed through Thorlabs OSA and Origin Pro software. Further performance analysis such as stability, repeatability and reversibility are utilized on optimized TOF sensor only. The stability measurement is taken every 5 minutes for 70 minutes period of time. For repeatability parameter, the TOF sensor reading will be taken in 3 cycles of time at lowest, middle and highest RI while reversibility is conducted by measuring the sensor response in ascending and descending order continuously.

There are some limitations applies in this study which include the mechanical tapering system that provides only single pulling direction mechanism. This condition causes the TOF experienced a non-adiabatic behaviour which define by uneven angles of up-taper and down-taper region. This particular obstacle caused the waist diameter of TOF cannot be fixed at precise value measurement.

1.5 Significance of Research

Recently, optical fiber sensor have been intensively explored in various application based on varying refractive index of the surrounding media. RI measurement in small volume plays significant role in biological areas which merge in physics, chemist and medicine studies such as in determining the concentration of proteins or glucose (17). Despite their popularity, implication of LSPR phenomena has been discovered due to its advantages in accuracy and high sensitivity behaviour. Proposed TOF incorporating sensitive gold nanolayer offers many advantages such as miniaturized structure, immunity to EM signals and the potential to be used in remote areas. In this context of study, the main goal is to rapidly detect even a small change of refractive indexes by optimize the coating parameter. Dedicated research and development of proposed sensor will be utterly useful for biochemical compound detection in real-time manners. Research findings will be used as the future reference in this particular field.

1.6 Research Outline

This thesis consists of five chapter which a brief outline of each chapter is given as follows.

Chapter 1 presents the introduction which included the background of study, problem statement, research objectives, research scope and significance of the study.

Chapter 2 includes the literature review on TOF configuration and its application based on LSPR response. Starts with optical fiber introduction then it follows by the existence of evanescent wave that contributed in sensor application is discussed. The mechanism of LSPR phenomena that present in sensing purpose also been elaborated in this section.

Chapter 3 starts with the research flow of the study under methodology section. Fabrication of TOF by flame brushing technique has been described. It follows by the deposition process on the TOF region surface by prepared AuNPs that synthesized by Turkevich method. The experimental setup has been illustrated and described.

Chapter 4 presents and discuss in details all results that obtained in this study. Deposition time which correspond to the coating thickness are determined and optimized. The surface morphology, nanoparticles characterization and LSPR response are analysed and discussed.

Chapter 5 provides the summaries of research outcomes in the study. Some recommendation has been suggested for further works.

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