# MODIFICATION OF INTRINSIC REFRACTIVE INDEX OPTICAL FIBER SENSOR VIA PRECISE CLADDING REMOVAL AND HETEROGENEOUS ZnO/Ag BI-LAYER COATING

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# DEDICATION

In the Name of Allah, the Most Merciful, the Most Compassionate all praise be to Allah, the Lord of the worlds. First and foremost, I must acknowledge my limitless thanks to Allah, the Ever-Magnificent; the Ever-Thankful, for His helps and bless. I am totally sure that this work would have never become truth, without His guidance. I dedicate my dissertation work to my family and my friends. A special feeling of gratitude to my loving parents (Ahmad and Hamideh) whose words of encouragement and push for tenacity ring in my ears and beloved brother (Alireza) who was like my backbone and still supports me virtually. He was the only reason for my current position. His love and dedication to the upliftment in my life is incredible. I pay homage to my brother. I am also dedicating this dissertation to my many friends who have supported me throughout the process. I will always appreciate all they have done, especially Mohamad Aizat Abu Bakar, Koo KhongNee, Dayang Norafizan Binti Awang Chee, Samuel Ansong and Vahid Khosravi.

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# ABSTRACT

Miniaturized refractive index sensor by combination of nanostructure thin films as a transformer sensitive layer and optical fiber as a signal carrier offers great potential for identifying the environmental features and understanding the novel sensor concepts. The partially unclad and bi-layer zinc oxide (ZnO) / silver (Ag) coated multimode glass and polymer optical fiber as a simple and reliable intrinsic fiber sensor was proposed in this work to detect the ambient refractive index changes (saline and crude oil having various concentrations) using two broadband sources of infrared radiation and ultraviolet-visible. The removing process to partially unclad the polymer and glass fiber was carried out precisely using our proposed dynamic monitoring process to prevent any interruption on propagating light by occurrence of damage on the core surface. The ZnO as an outer sensitive layer had three configuration which was spherical nanoparticle, horizontally and vertically oriented nanorods deposited on discontinuous Ag layer using mixture of electroless, dip coating and low temperature hydrothermal techniques because solo deposition technique was not possible. Ag nano-island shape deposition made the transition of evanescent wave to external media possibly through this semi-reflectance structure. ZnO coating avoided the formation of oxygen deficit defects, inhibited aging problem and trapping measurand molecules through mechanical interlock phenomena which altered its optical characteristics and improved sensitivity of the sensor. The x-ray diffraction spectra demonstrate that the level of crystallinity was higher for vertically oriented ZnO compared to others. Using field emission scanning electron microscope images, the width/length of vertically and horizontally oriented ZnO nanorods was measured to be ~ 86 nm/~ 690 nm and ~ 67 nm/~ 544 nm, respectively. The ZnO nanoparticle size was in the range of ~ 10 nm to ~ 75 nm. Surface roughness of ZnO/Ag coated glass (polymer) probe extracted from atomic force microscopy was ~ 39 (52 nm), ~ 52 (176 nm) and ~ 148 (346 nm) for nanoparticles, horizontally and vertically nanorods respectively. Room temperature Photoluminescence spectra from bi-layer ZnO/Ag coated on glass substrate when contacted with saline and crude oil having different concentrations revealed that near band edge emission band gap shifted from ~3.447 eV to ~3.189 eV going from sphere nanoparticles to vertically oriented nanorods. This shift is independent with the contact media. However, deep level emission depends strongly on the concentration of the contacted media. The shift observed for nanoparticle, horizontally and vertically oriented ZnO/Ag when contacted with saline (crude oil) was ~ 0.05 eV (~ 0.09 eV), ~ 0.02 eV (~ 0.03 eV) and ~ 0.08 eV (~ 0.11 eV) respectively. The performance of fabricated probe to detect saline concentration changes for glass fiber coated with vertically oriented ZnO nanorods/Ag when IR light source employed, was supreme compared to the other samples and was reported to be 255.4 nm/RIU and 314.2 dB/RIU for wavelength and intensity sensing respectively. The durable polymer fiber coated with vertically oriented ZnO/Ag nanorods showed the intensity and wavelength sensitivity of 146.2 dB/RIU and 78.5nm/RIU respectively in identifying the variation of crude oil from 0 to 100%. The optimum length of glass and polymer fiber probe for maximum sensitivity was obtained for 3 cm and 2 cm respectively. The precise production techniques together with comprehensive analysis of the sensing mechanism lead to a deeper understanding of the liquid refractive index behavior applicable in quality control in water resource and oil reservoir.

# ABSTRAK

Penderia indeks biasan miniatur dengan gabungan filem nipis nanostruktur sebagai lapisan sensitif pengubah dan gentian optik sebagai pembawa isyarat berpotensi untuk mengenal pasti keadaan sekeliling dan memahami konsep penderia baharu. Gentian optik kaca dan polimer berbilang mod yang telah dinyahsalut sebahagiannya dan disalut dengan dwilapisan zink oksida (ZnO) / perak (Ag) bertindak sebagai penderia gentian intrinsik yang mudah dan boleh dipercayai dicadangkan untuk mengesan perubahan indeks biasan (air garam dan minyak mentah pada pelbagai kepekatan) dengan menggunakan dua sumber jalur lebar, inframerah dan ultraungu-cahaya tampak. Proses penyahsalutan separa gentian polimer dan kaca secara terperinci dilakukan dengan pemantauan secara dinamik seperti yang dicadangkan untuk mengelakkan sebarang gangguan terhadap penyebaran cahaya melalui kerosakan pada permukaan teras gentian optik. ZnO sebagai lapisan sensitif luaran mempunyai tiga konfigurasi iaitu nanopartikel sfera, nanorod berorientasi mendatar dan menegak, endapan pada lapisan Ag yang berlainan menggunakan campuran elektroless, salutan celupan dan teknik hidrotermal suhu rendah karana teknik endapan tidak dapat dilakukan secara solo. Endapan nano-pulau Ag menjadikan peralihan gelombang sisihan ke media luaran terhasil melalui struktur pemantulan separa ini. Salutan ZnO mengelakkan pembentukan kerosakan defisit oksigen, merencat masalah penuaan dan memerangkap molekul ukuran melalui fenomena interlok mekanikal yang mengubah percirian optik dan meningkatkan sensitiviti penderia. Spektrum pembelauan sinar-X menunjukkan bahawa tahap pengkristalan ZnO yang berorientasi tegak lebih tinggi berbanding yang lain. Melalui imej mikroskop imbasan elektron pancaran medan, lebar / panjang nanorod ZnO yang berorientasikan secara menegak dan melintang diukur masing-masing sebanyak ~ 86 nm / ~ 690 nm dan ~ 67 nm / ~ 544 nm. Saiz nanopartikel ZnO ialah dalam julat ~ 10 nm hingga ~ 75 nm. Kekasaran permukaan kuar kaca (polimer) bersalut ZnO / Ag yang diekstrak dari mikroskop daya atom masing-masing adalah ~ 39 (52 nm), ~ 52 (176 nm) dan ~ 148 (346 nm) untuk nanopartikel, secara mendatar dan menegak. Spektrum foto pendarcahaya pada suhu bilik dari lapisan ZnO / Ag bersalut pada substrat kaca apabila bersentuhan dengan larutan garam dan minyak mentah yang berbeza kepekatan menunjukkan peralihan hampir jalur pinggir ~ 3.447 eV ke ~ 3.189 eV nanopartikel sfera ke arah nanorod berorientasi tegak. Peralihan ini tidak dipengaruhi oleh media sentuhan. Walau bagaimanapun, pelepasan paras dalam sangat bergantung kepada kepekatan media yang bersentuhan. Peralihan yang diperhatikan untuk nanopartikel, ZnO / Ag berorientasikan secara mendatar dan menegak apabila berinteraksi dengan larutan garam (minyak mentah) masing-masing adalah ~ 0.05 eV (~ 0.09 eV), ~ 0.02 eV (~ 0.03 eV) dan ~ 0.08 eV (~ 0.11 eV). Prestasi kuar yang difabrikasi untuk mengesan perubahan kepekatan garam untuk gentian kaca yang dilapisi dengan nanorod berorientasikan ZnO / Ag menegak ketika sumber cahaya IR digunakan, dibandingkan dengan sampel yang lain dan dilaporkan masing-masing sebanyak 255.4 nm / RIU dan 314.2 dB / RIU untuk panjang gelombang dan intensiti. Polimer tahan lasak yang bersalut dengan nanorod ZnO / Ag berorientasikan menegak menunjukkan sensitiviti intensiti dan panjang gelombang sebanyak masing-masing 146.2 dB / RIU dan 78.5 nm / RIU untuk mengenal pasti kepekatan minyak mentah dari 0% hingga 100%. Panjang optimum kuar gentian kaca dan polimer untuk sensitiviti maksimum adalah masing-masing pada 3 cm dan 2 cm. Teknik pembuatan yang teliti berserta dengan analisis yang komprehensif membawa kepada kefahaman yang lebih mendalam terhadap keadaan serakan indeks cecair yang mana mampu menyumbang kepada kawalan mutu sumber air dan takungan minyak.

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| | | |
|---|---|---|
| AFM | - | Atomic Force Microscopy |
| ALD | - | Atomic Layer Deposition |
| CEI | - | Cladding Environment Interface |
| CVD | - | Chemical Vapor Deposition |
| DC | - | Direct Current sputtering |
| DI | - | Distilled Water |
| DL | - | Deep-Level |
| EDX | - | Energy Dispersive X-ray Spectroscopy |
| EMI | - | Electro Magnetic Interference |
| Eq | - | Equation |
| EW | - | Evanescent Wave |
| Exp | - | Experimental |
| FBG | - | Fiber Bragg Grating |
| FESEM | - | Field Emission Scanning Electron Microscopy |
| FWHM | - | Full Width at Half Maximum |
| SG | - | Specific Gravity |
| IEA | - | International Energy Agency |
| USGS | - | US geological survey |
| GFiber | - | Glass Fiber |
| GOF | - | Glass Optical Fiber |
| HOMO | - | Highest Occupied Molecular Orbital |
| JCPDS | - | Joint Committee on Powder Diffraction Standards |
| LUMO | - | Lowest Unoccupied Molecular Orbital |
| LPG | - | Long Period Grating |
| LSP | - | Localized Surface Plasmon |
| LSPR | - | Localized Surface Plasmon Resonance |
| LSPRs | - | Localized Surface Plasmon Resonance Sensor |
| MBE | - | Molecular Beam Epitaxy |
| MOCVD | - | Metal Organic Chemical Vapour Deposition |
| NBE | - | Near Band Edge |

| | | |
|---|---|---|
| NPs | - | Nanoparticles |
| NRH | - | Nanorods Horizontally |
| NRV | - | Nanorods Vertically |
| OSA | - | Optical Spectrum Analyzer |
| PC | - | Poly Carbonate |
| PVD | - | Physical Vapor Deposition |
| PECVD | - | Plasma Enhanced Chemical Vapour Deposition |
| PFiber | - | Polymer Fiber |
| PL | - | Photoluminescence |
| PMMA | - | Poly (methyl methacrylate) |
| POF | - | Plastic Optical Fiber |
| PS | - | Poly Styrene |
| RF | - | Radio Frequency Sputtering |
| RI | - | Refractive Index |
| RT | - | Room Temperature |
| SEM | - | Scanning Electron Microscopy |
| SPR | - | Surface Plasmon Resonance |
| TEM | - | Transverse Electro Magnetic |
| Theo | - | Theory |
| TIR | - | Total Internal Reflection |
| UV-VIS/NIR | - | Ultraviolet-Visible/Near Infrared |
| XRD | - | X-Ray powder Diffraction |
| ZP | - | Zeta Potential |

## LIST OF SYMBOLS

| | | |
|---|---|---|
| $\mu m$ | - | Micrometer |
| nm | - | Nanometer |
| dBm | - | Decibel-milliwatts |
| mW | - | Milliwatts |
| mA | - | Milliampere |
| eV | - | Electron volt |
| mV | - | Millivolt |
| ppm | - | Parts-per-million |
| cm | - | Centimeter |
| min | - | Minute |
| °C | - | Celsius degree |
| W/V | - | Weight per volume |
| V/V | - | Volume per volume |
| RH | - | Relative humidity |
| E | - | Evanescent field |
| $d_p$ | - | Penetration depth |
| $\theta$ | - | Incident angle of light |
| $\lambda$ | - | Wavelength |
| $\lambda_0$ | - | Wavelength of incident light |
| $dp$ | - | Penetration depth of the evanescent field |
| n | - | Refractive index |
| $\lambda_B$ | - | Bragg grating wavelength |
| ɵ | - | Angle of incidence normal at the interface |
| $n_{eff}$ | - | The effective reflective index of the fiber core |
| $E_{Total}$ | - | Total cohesive energy |
| $E_0$ | - | Cohesive energy of the bulk material |
| N | - | Number of surface atoms |
| A | - | Avogadro's number |
| $\sigma_0$ | - | Pre-exponential constant |
| $E_c$ | - | Energy of conduction band |

| | | |
|---|---|---|
| $E_F$ | - | Fermi energy |
| $T_{mn}$ | - | Melting temperature of nanosize materials |
| $T_{mb}$ | - | Melting temperature of bulk materials |
| $\Delta E_g$ | - | Energy bandgap variance |
| D | - | Diameter of spherical nanoparticle |
| l | - | Diameter of nanorod |
| $\xi$ | - | Absorption coefficient |
| $R_r$ | - | Roughness |
| $D_i$ | - | Local diameter |
| $h_i$ | - | Local depth |
| rf | - | Radio frequency |
| dc | - | Direct current |
| $\varphi$ | - | Phase difference |
| $\varepsilon$ | - | Dielectric constant |
| $K_{sp}$ | - | Propagating constant of the surface plasmon |
| $K_{e\omega}$ | - | Propagating constant of the evanescent wave |
| $\alpha$ | - | Absorption coefficient |
| $\omega$ | - | Angular frequency |
| $Q_{SPR}$ | - | Quality factor surface plasmon resonance |
| I | - | Intensity |
| $\alpha$ | - | Absorption coefficient |
| $t$ | - | Film thickness |
| D | - | Domain size |
| ß | - | Full width |
| $\gamma$ | - | Evanescent absorption coefficient |
| P | - | Power transmitted |
| $\rho$ | - | Core radios |
| N | - | Total number of nanoparticles |
| Ag | - | Silver |
| Ag NPs | - | Silver nanoparticles |
| Au NPs | - | Gold nanoparticles |
| ZnO | - | Zinc oxide |
| Co | - | Cobalt |

| | | |
|---|---|---|
| $N_2$ | - | Nitrogen |
| HF | - | Hydrofluoric acid |
| Pd | - | Palladium |
| NaCl | - | Sodium chloride |
| GO-ZnO | - | Zinc oxide nanoparticle incorporated grapheme oxide |
| Ni | - | Nickel |
| $AgNO_3$ | - | Silver nitrate |
| $NH_3$ | - | Ammonia solution |
| NaOH | - | Sodium hydroxide |
| $Zn(NO_3)_2 6H_2O$ | - | Zinc Nitrate hexahydrate |
| $SnCl_2$ | - | Tin (II) chloride |
| $PdCl_2$ | - | Palladium (II) chloride |
| $(CH_3)_2NHBH_3$ | - | Boron hydride dimethylamine |
| HCl | - | Hydrochloric acid |
| $H_2SO_4$ | - | Sulfuric acid |
| Ge | - | Germanium |
| $GeO_2$ | - | Germanium dioxide |
| $P_2O_5$ | - | Phosphorus pentoxide |
| F | - | Fluorine |
| $B_2O_3$ | - | Boron trioxide |
| O | - | Oxygen molecule |
| Si | - | Silicon |
| $SiO_2$ | - | Silicon dioxide |
| $SiF_4$ | - | Silicon tetrafluoride |
| $SiF_6$ | - | Hexafluorosilicate |
| $Ag_2O$ | - | Silver Oxide |
| $NH_3$ | - | Ammonia |
| $Ag(NH_3)_2$ | - | Di ammine silver(I) |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Optical fiber sensor is a device that exchanges the light rays into electronic signals and it has connected to a light source to allow the detection of modulated light. It measures the physical changes in the amount of light and translates it into a form of signal which is readable by the optical spectral analyzer. A multiple sensing application have been revolutionized by representing the fiber optic sensors. Possibility of preparing a variety of fiber sensing structures by glass and polymer optical fiber makes them an ideal candidate for fabrication of sensing device. Fiber optic sensors are generally classified as either intrinsic or extrinsic. In the intrinsic sensor, the physical properties of light inside the fiber are modulated by the physical detecting parameter whereas in an extrinsic sensor, light modulation occurs outside the fiber (Liaw, 2019). In the former, there is possibility to modulate one of the physical properties of the guided light such as wavelength, intensity, polarization and phase by the measurand. In the latter case, the fiber only acts as channel to transport the light signal to and from the sensor probe. However, out of these four properties of light, the intensity and wavelength modulated ones propose the widest range of optical fiber sensors (Du et al., 2019; Wang et al., 2018; Efendioglu, 2017). The special characteristics of being non-electrical, small in size, rugged and immune to electromagnetic interference boosts the use of optical fibers for sensing applications in the field of engineering, science and technology (Mowbray et al., 2019; Güemes, 2014). The physical and chemical properties such as temperature (Guo et al., 2019), liquid level (Díaz et al., 2019), radiation (Mikel et al., 2019), strain (Nascimento et al., 2019), refractive index (Gowri et al., 2019), vibration (Kuribayashi et al., 2019), concentration of liquid (Wang et al., 2019) and chemical analysis (Kaushik et al., 2019) can be detected by optical fiber sensors. Furthermore, low loss, low dispersion, ultra-wide bandwidth, high dynamic range, durability and upgradability are the other reasons for shifting the attention from traditional electrical sensors to optical fiber sensors (Barrias et al., 2016). Compatibility to the multimode fiber technology and simplicity of construction are two main advantages of intensity and wavelength modulated sensors (Bag et al., 2020; Zhang et al., 2020).

## 1.2 Statement of the Problems

For developing the optical fiber sensor, the diameter of optical fiber must be reduced by removing the cladding part. Due to refraction of evanescent wave in the cladding and its absorption in surrounding media, the etched region of optical fiber becomes more sensitive (Korposh et al., 2019). For removing the cladding of optical fiber, several ways are existed. The current techniques for stripping the cladding can be divided into mechanical and chemical methods. Polishing the fiber is the most common mechanical technique to remove the cladding (Addanki et al., 2018). However, significant disadvantage of this method is that the fiber optic stripper can potentially damage the fiber core and it typically requires more expensive equipment. Furthermore, lasers and precision lenses on laser processing platforms with a moving mechanism are used for removing purpose, however, the precision lens on the laser processing platform tends to age, which may affect the accuracy of the moving platform and lead to cause experimental errors (Lin et al., 2019). Moreover, the problems such as an inability to correctly remove materials, and/or changes in the material properties may occur using the laser (Pospori et al., 2017). The chemical method like using of various solutions such as Hydrofluoric acid (HF) for glass optical fiber (GOF) and organic solvents for polymer optical fiber (POF) was employed by different researchers (Zaca-Morán et al., 2018; Razzaq et al., 2020; Subashini et al., 2018; Inglev et al., 2019). However, the etching process is difficult to control because a slight error can generate unexpected processing like damaging the core outer layer as a result affect the light propagating and sensing quality. Regardless of which method is employed, they all provide a lack of comprehensive post-processing quality control procedures.

It was discovered that the sensitivity of optical fiber sensors depends on the radius of the fiber, taper waist length, launch angle and surface roughness of the sensing area (Qazi et al., 2019; Rajamani et al., 2019; Qaziet al., 2019; Ma et al., 2019). Despite the fact that the light-scattering loss is increased by increasing the surface roughness, the sensors with rough surfaces exhibit higher sensitivity than those with smooth surfaces (Sequeira et al., 2019). The effects of core roughness after fully etching the cladding, on performance of conventional core-clad structure glass fiber sensors have previously been studied (Liu et al., 2002; Kimet al., 2010; Leal-Junior et al., 2018; Qazi et al., 2019), but the effects of cladding roughness associated with varying solvent concentration on sensitivity of partially unclad optical fiber probe have not been investigated extensively. Cladding thickness plays a crucial role on sensitivity of the sensor therefore, the removed cladding part in micrometer scale have been reported elsewhere. However, systematic and accurate control of cladding removal in nanometer have not been reported.

Currently, Ag coated optical fiber probe due to superior properties of Ag such as surface plasmon resonance (SPR), electron donor in dark and high reflectivity in the visual to near-infrared region has been attracted huge attention for measuring the refractive index changes (Fu et al., 2019; Lee et al ., 2018; Shen et al., 2008). However, there is some backwards of using this material. Aging and exposing to atmosphere cases the oxygen deficit defects formed the silver oxide matrix on the sensing layer which consequences to shortening the life time and lowering the sensitivity of the fiber optic (Lee et al., 2018; Jiu et al., 2015). Moreover, high reflectivity leads to reflect the evanescence wave back completely to the core and prevent contacting with media.

Glass and polymer optical fiber as nonconductive substrates can be coated by chemical and physical vapor deposition (CVD and PVD), chemical electroless plating, sol gel and chemical bath deposition. However, continuous deposition type, high-temperature treatments and huge energy supply systems (Christopher et al., 2018; Ozcariz et al., 2019) which are required for PVD and CVD make them unsuitable for our purpose. In sol-gel method there is a little control over porosity of the gel which in turn affects the rate of solvent removal from the gel in order to form the final powder and similar to the other methods, discontinuous coating also is not possible (Tang et al., 2017). Electroless plating is a promising technique for uniform metallic coating where discontinuous deposition would be possible however, deposition of different nanostructure configuration is not provided by this method. Moreover, it should be mentioned that in this study both glass and polymer fiber is employed to fabricate sensor due to extending their application and considering their pros and cons once using them in various devices and environment.

## 1.3 Objectives of the Study

Considering the research background and the problem statements mentioned before, the main objective of this study is to fabricate sensitive optical fiber probe to detect the refractive index variance of different liquids. The specific objectives are shown as follow:

a) To fabricate partially unclad glass and polymer optical fiber sensors.

b) To modify partially unclad optical fiber probe via ZnO/Ag bi-layer coating having ZnO nanoparticles and nanorods as an outer layer and investigate the influence of bi-layering on sensitivity.

c) To evaluate and optimize the sensing parameters including shape of the zinc oxide nanostructure, length of the probe and propagating wavelength on sensor performance.

## 1.4 Scopes of the Research

For achieving the above stated objectives, following scopes of works have been presented:

a) Preparing partially unclad glass and polymer multimode, step index optical fiber sensors for refractive index changes detection the following steps are performed:

i. Removing the part of cladding using dilute hydrofluoric acid in the range of 35-20 % with decreasing ratio of 5% for glass fiber and mixture of acetone/methanol in the range of (50/50), (40/60), (30/70) and (20/80) for polymer fiber. For both fibers the temperature varies from 15 to 30 °C by increasing ratio of 5 °C.

ii. Measuring the diameter of cladding using field emission scanning electron microscopy (FESEM) to observe the remain cladding thickness and its configuration. Atomic force microscopy (AFM) to obtain the cladding surface roughness. Dynamic monitoring system including solvents, broad band light source with a laser wavelength ranges from 360 to 2600 nm and power meter as a detector are employed. Higher (crude oil) and lower (saline) refractive index solutions with concentration of 0 to 100% and 0 to 20% are used respectively. The refractive index of the solution is measured by refractometer.

b) For coating the fiber with ZnO/Ag bi-layer nanostructures and study their features following steps are carried out:

i. Electroless deposition technique is used for preparing the Ag nano-layers. The mixture of electroless, dip coating and low temperature hydrothermal method are employed for zinc oxide nanostructure deposition on top of the Ag layer.

ii. Using field emission scanning electron microscopy (FESEM) the nanostructures size is measured and their shapes are observed. Energy dispersive X-ray spectroscopy (EDX) confirms the formation of deposited layer and examines the elemental distribution of the sample. X-Ray diffraction spectroscopy (XRD) estimates the degree of crystallinity and illustrates the structure of the deposited materials. Atomic force microscopy (AFM) reveals the morphology, size, roughness and surface topography of the fabricated probe. Contact angle (CA) is used to characterize the wettability of the probe surface. Zeta potential (ZP) is employed to determine the electrochemical surface properties and existence of charges on the surface. Photoluminescence (PL) spectroscopy determines the optical properties including the electronic bandgap, crystal defect energy level and changes the nanostructure band gap once exposing to different refractive index media. The UV-Vis/NIR transmission and absorption are employed for further optical characterization. Tensile test is carried out to measure the physical properties of the probe and its despondence to the applied stress. Refractometer measures the refractive index of the saline and crude oil solutions. Finally, the ImageJ program is applied to determine the dimension, size, and distribution of the ZnO nanostructure deposited on top.

c) For fulfilling the third objective regarding to optimization of sensing parameters the following process is carried out.

i. Light sources of UV-Vis and IR (ranges from 360 nm to 2600 nm) are employed. The optical spectrum analyzer (OSA) is used as a light detector and multimode optical fibers having sensing length of 1 to 5 cm are applied. Saline and Crude oil solutions with different volume concentrations (ranges from 0 to 20% and 0 to 100%) are prepared as the measurand liquids.

## 1.5 Significance of Study

Nanosizing of materials is prerequisite for developing new electronic and optoelectronic devices. Especial miniature optical fiber sensors have sensitive thin films as a probe that can open new field in optical fiber sensor applications. Optical fibers act as signal carrier and thin films work as sensitive elements and transducer to get response and feedback from environments. The utilization of a highly sensitive, flexible, low cost, and small size intrinsic optical fiber sensor based on exterior cladding modifications for detecting the crude oil and saline concentration, permits operation at harsh environment with remote sensing operation capability, where bulk extrinsic sensors is not suitable to use. The magnitude of the salinity changes is a critical factor for determining the chemistry of natural waters and biological processes. The label-free refractive index sensor is promising device for detecting these changes. Therefore, an accurate monitoring of concentration changes in saline solution is prerequisite to control and minimize the negative effect of salt in water resources. Furthermore, efficient and accurate estimation of crude oil density changes is an essential factor in reservoir engineering. Determining the refractive index gradient as a representative of these changes by optical fiber sensor offers a novel approach in oil production optimization.

A novel ZnO/Ag bi-layer coated intensity and wavelength modulated optical fiber sensors having variety of ZnO shapes based on refractive index changes using IR and UV-Vis light sources are proposed. It is believed that this is the first work that the economic electroless technique as a promising chemical method is used for bi-layer deposition of materials on multimode optical fiber. Moreover, controlling the ZnO nanostructure shape which has direct effect on sensing mechanism can be controlled by chemical techniques and has not been extensively done. The possibility of tuning the optical response of coated nanomaterials by modifying their cladding part, synthesize techniques, length of the sensing area, nanostructure configuration and applying different light source has become one of the most challenging aspects of recent fiber optic sensor research and have been successfully fulfilled in this thesis.

For controlling the assembly process and maintaining the quality of final products in manufacturing development where remote sensing is demanded, our organized fabricated micro scale sensor with low disturbance and without any explosion risks can contribute more effective than the other types of sensors. The proposed fabrication technique would be easy and economic. Large-scale and socio-economic instrumentation is provided. The fundamental phenomena and details of sensing mechanism would be fully understood. The device fabricated by this research can be used in wide range of industries and the data created will be published in high impact factor journal and presented in workshops, conferences and seminars. This methodology can be used to train the PhD and masters research scholar. High quality home fabricated optical fiber sensors can support the demands in optoelectronic industries. Measuring the small changes in refractive index would be possible by a set of characterization that is proposed. A right fiber optic sensor configuration for refractive index monitoring will be offered. These methodologies are not just limited to the deposited silver and zinc oxide nanostructures and it can be extended to other materials based on application needed. Other nanostructure materials like gold, nickel, copper and other also can be used as sensing part.

## 1.6 Organization of Thesis

This thesis structurally is divided into five chapters giving a complete fabrication, characterization and performance on partially unclad optical fiber sensors coated with bi layer ZnO/Ag for developing optical fiber sensors to monitor refractive index (concentration) of saline and crude oil solutions. The current chapter presents a short introduction on fiber optics field that consist of the motivation and the objectives of this research.

Chapter 2 provides more information of an available literature review. This chapter consists of the theory of sensing mechanism and classification of optical fiber probe considering different aspects. Moreover, the feature and potential of ZnO and Ag as sensitive coated materials are extensively discussed. Varieties of deposition techniques are listed and among them electroless method is explained.

Chapter 3 includes a description of the fabrication of glass and polymer partially unclad optical fiber sensors based on solvent concentration and temperature. Moreover, fabricating of polymer and glass probe using deposition of bi-layer ZnO/Ag having different configuration of ZnO on partially unclad optical fiber via mixture of three techniques of electroless, dip coating and low temperature hydrothermal method are explained. Finally, background information of major experimental tools or techniques for collecting data is explained.

In chapter 4 the fabricated partially unclad optical fiber is characterized to view the morphology, structure and its performance. The role of probe length and etching solution concentration discussed in detail and the results are presented. Then, the optical features of ZnO/Ag nanostructures are investigated. The performance of ZnO/Ag bi-layer coated on polymer and glass fiber having variety of top-layer configurations is studied and presented. The sensitivity comparison for all fabricated probe is carried out and listed. Physical characteristic of the polymer and glass fiber sensor fabricated with optimum parameters is studied.

Chapter 5 gives a summary and review of the results and analysis of this study. It additionally includes limitation of this research and suggestions for further research work. Attached in the appendix contain published papers during this study.

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## LIST OF PUBLICATIONS

### Journal with Impact Factor

1. **Samavati, Z**., Samavati, A., Ismail, A.F., Yahya, N., Rahman, M.A. and Othman, M.H.D. (2020). Effect of acetone/methanol ratio as a hybrid solvent on fabrication of polymethylmethacrylate optical fiber sensor. *Optics & Laser Technology*, 123, p.105896. **(Q1, IF: 3.323)**
2. **Samavati, Z**., Samavati, A., Ismail, A.F., Yahya, N., Othman, M.H.D. and Rahman, M.A. (2020). Modified polymer optical fiber sensors for crude oil refractive index monitoring. *Journal of Materials Science: Materials in Electronics*, pp.1-12. **(Q2, IF: 2.220)**
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**8.** Samavati, A., **Samavati, Z**., Ismail, A.F., Yahya, N., Bakar, M.A.A., Othman, M.H.D., Rahman, M.A., Koo, K.N. and Osman, S.S. (2019). Magnetic field detection using a highly sensitive FBG probe. *Physica Scripta*. **(Q2, IF: 1.985)**

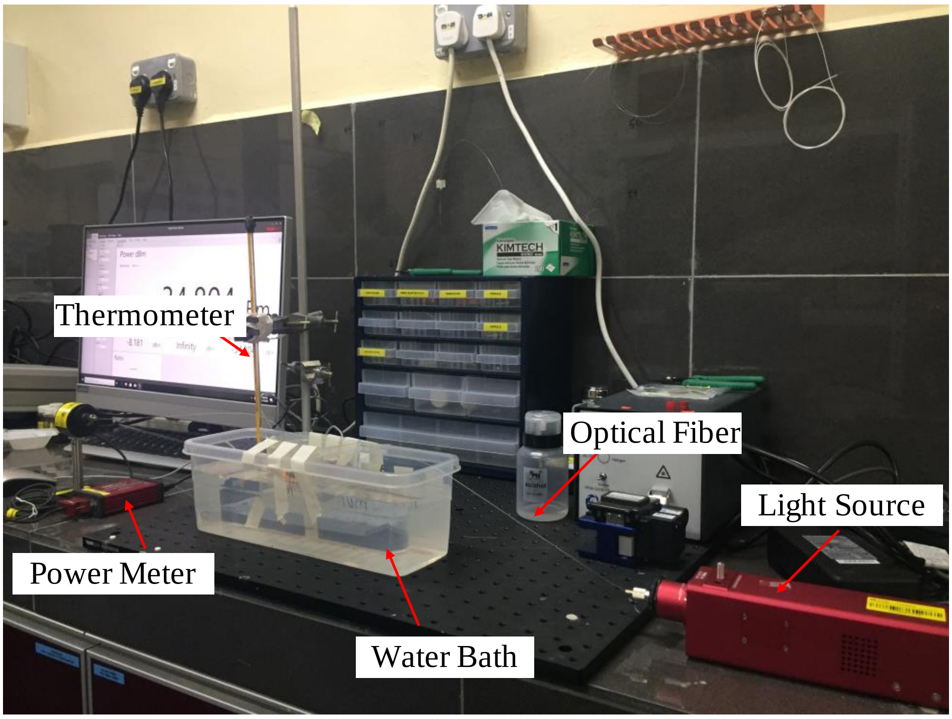
**9.** Samavati, A., **Samavati, Z**., Ismail, A.F., Yahya, N., Othman, M.H.D., Rahman, M.A., Bakar, M.A.A., Koo, K.N., Salebi, M.F. and Amiri, I.S. (2019). An FBG magnetic sensor for oil flow monitoring in sandstone core. *RSC Advances*, 9(61), pp.35878-35886. **(Q2, IF: 3.119)**

**10.** Samavati, A., **Samavati, Z**., Ismail, A.F., Othman, M.H.D., Rahman, M.A. and Amiri, I.S. (2018). Effect of organic ligand-decorated ZnO nanoparticles as a cathode buffer layer on electricity conversion efficiency of an inverted solar cell. *RSC advances*, 8(3), pp.1418-1426. **(Q2, IF: 3.119)**

**International Conferences**

1. **Samavati, Z**., Ismail, A.F. and Rahman, M.A. (2018). ZnO/Ag/Glass optical fiber as a sensing probe for detecting the refractive index changes. *National congress of membrane technology*. 30th-31st October 2018, Johor Bahru, Malaysia.
2. **Samavati, Z**., Samavati, A., Ismail, A.F., Rahman, M.A. and Othman, M.H.D. (2019) Optical Fiber Refractive Index Sensor Based on Partially Unclad Multimode Fiber Coated with ZnO Nanoparticles. *International Conference of Sustainable Environmental Technology*. 20th-22nd August 2019, Johor Bahru, Malaysia.
3. **Samavati, Z**., Samavati, A., Ismail, A.F., Yahya, N., Rahman, M.A. and Othman, M.H.D. (2019) The role of acetone concentration as etching solvent on fabrication of partially unclad polymer optical fiber sensor. *International Laser Technology and Optics Symposium 2019*. 3rd – 4th September 2019, Johor Bahru, Malaysia.

# APPENDIX A



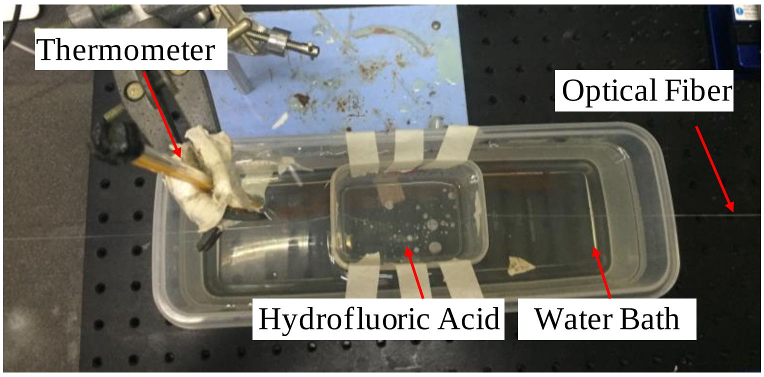




Figure A.1 Un-cladding process via etching method by varying the temperature and solvents concentrations for GOF and POF.

**APPENDIX B**

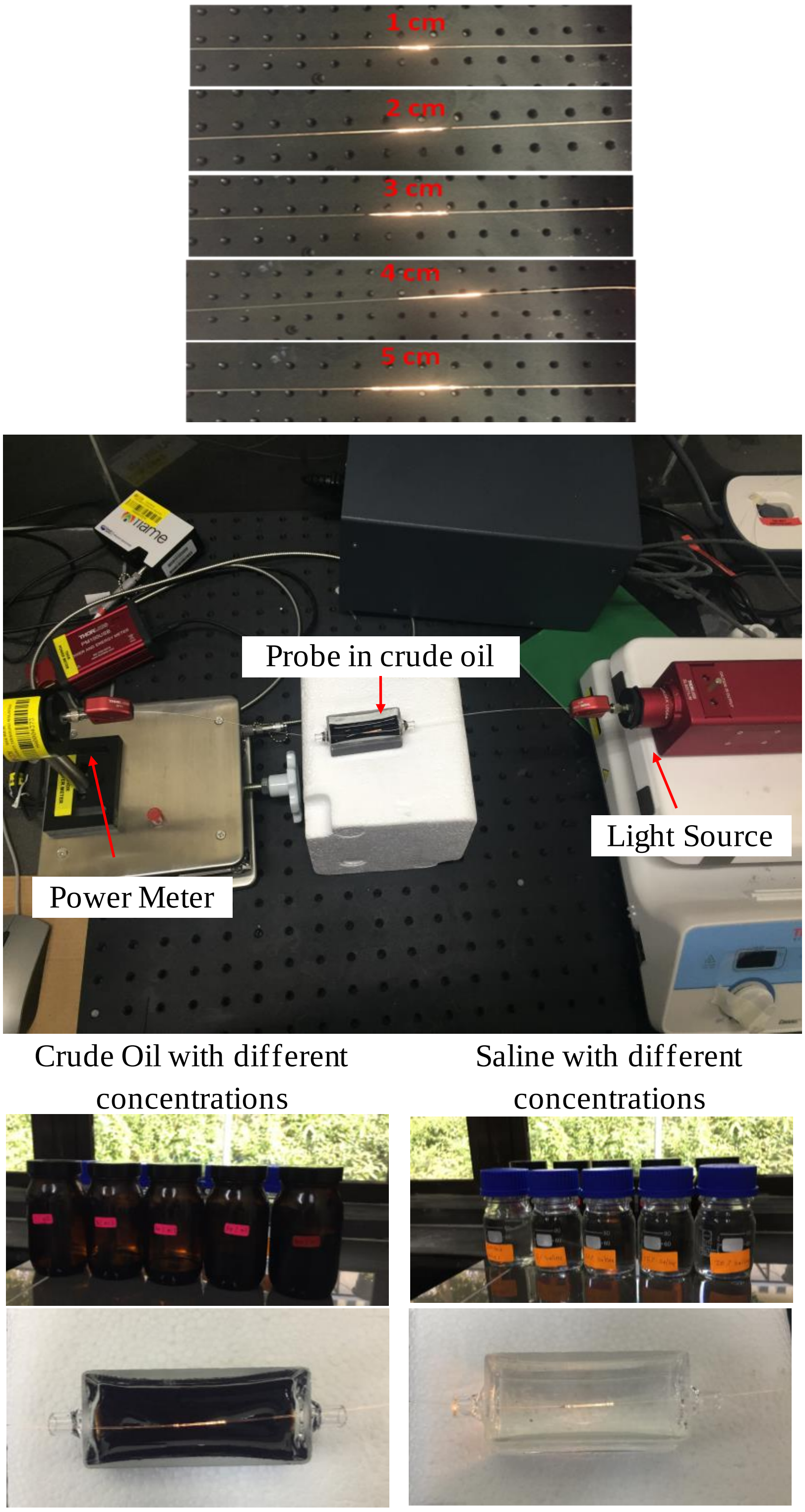



Figure B.1 performance analysis of partially unclad fiber against refractive index changes. The refractive index of saline and crude oil solutions varied from 1.333-1.368 and 1.372-1.478 respectively.

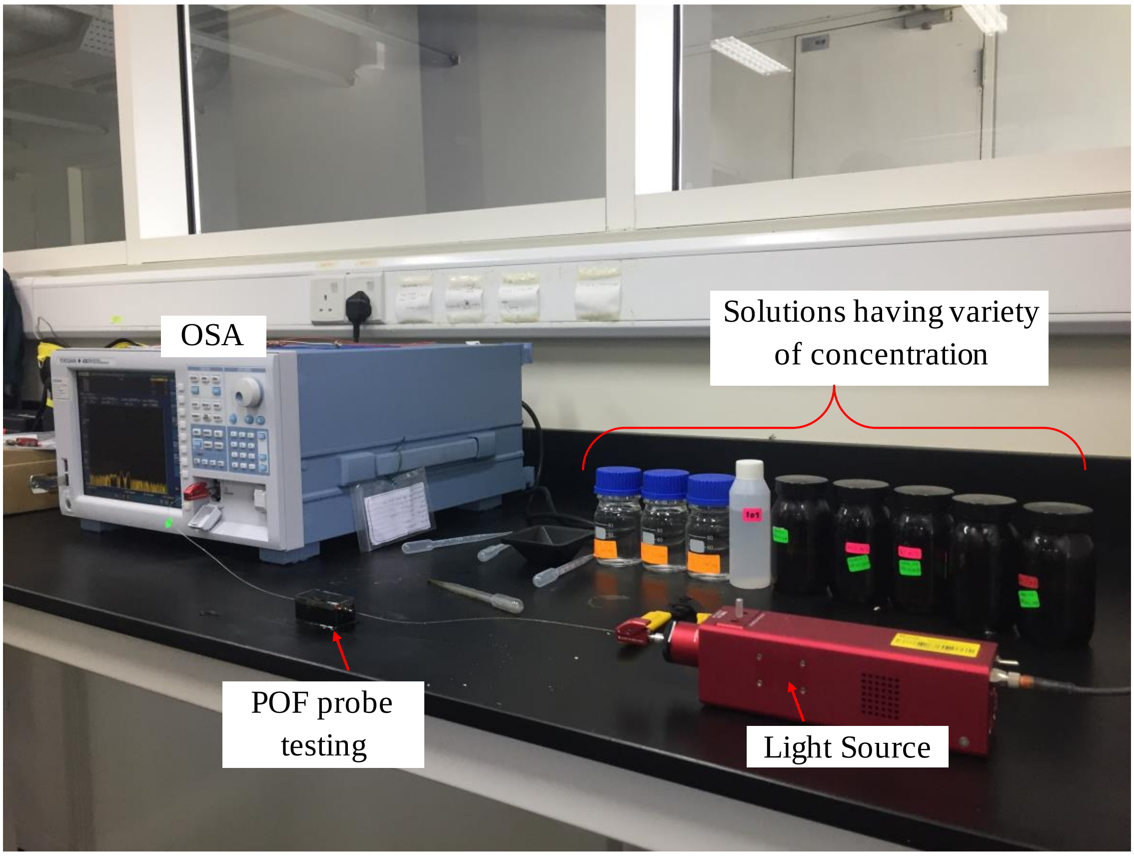



Figure B.2 performance process of probes coated with bi- layer ZnO/Ag on POF and GOF for detecting the concentration changes of saline and crude oil.