STRUCTURED GROWTH OF ZINC OXIDE NANORODS
ON PLASTIC OPTICAL FIBER AND LIGHT SIDE
COUPLING TOWARDS SENSING APPLICATIONS

HAZLI RAFIS BIN ABDUL RAHIM

FACULTY OF ENGINEERING
UNIVERSITY OF MALAYA
KUALA LUMPUR

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HAZLI RAFIS BIN ABDUL RAHIM

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Name of Candidate: Hazli Rafis Bin Abdul Rahim (I.C No.: 811110-02-5401)
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Field of Study: Electronic (Engineering and Engineering Trades)

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ABSTRACT

A simple and cost effective optical fiber sensor using side coupling of light into the core modes of plastic optical fiber (POF) coated with zinc oxide (ZnO) nanorods is reported here. Nanorods coating enhanced coupling inside the fiber by scattering light but were also capable of causing leakage. Structuring the growth to specific regions allowed scattering from different segments along the fiber to contribute to the total coupled power. A uniform, densed and highly aligned spiral patterned ZnO nanorods were grown on the POF using the hydrothermal method and its effect was investigated. ZnO nanorods growth time of 12 h and temperature of 90 °C provided the best coupling voltage. Side coupling was measured to be a factor of 2.2 times better for spiral patterned coatings as opposed to unpatterned coatings. The formation of multiple segments was used for multiple-wavelength channels excitation where different bands were side coupled from different segments. It was found that visible white light source significantly coupled the light into the POF compared with infrared laser sources. A first order theoretical model was derived to simulate the impact of millimeter (mm) scale spiral patterns on coupling efficiency by varying the width and spacing of the coated and uncoated regions. The width of spiral patterned ZnO nanorod coatings on POF was optimized theoretically for light side coupling and was found to be 5 mm. An experimental validation was performed to complete the optimization and the experimental results showing a well correlation with simulation. Optimized width of spiral patterned ZnO nanorods grown on large core POFs was used for the purpose of temperature and multiple optical channel alcohol vapor sensing. Spiral patterned ZnO nanorods coating exhibited a significant response to temperature change from 20 °C to 100 °C based on extinction concept which is the attenuation of light by scattering and absorption as it traverses the ZnO nanorods. Sensitivity was measured to be a factor of 1.3 times better for spiral patterned coatings as opposed to unpatterned coating. The multiple optical channel alcohol sensing mechanism
utilized changes in the output signal due to adsorption of methanol, ethanol and isopropanol vapors. Three spectral bands consisting of red (620-750 nm), green (495-570 nm) and blue (450-495 nm) were applied in measurements. The range of relative intensity modulation (RIM) was determined to be between 25 to 300 ppm. Methanol presented the strongest response compared to ethanol and isopropanol in all three spectral channels. With regard to alcohol detection RIM by spectral band, the green channel demonstrated the highest RIM values followed by the blue and red channels respectively.
ABSTRAK

Satu penderia optik yang mudah dan kos efektif menggunakan gandingan sisi cahaya ke dalam ragam-ragam teras gentian optik plastik (POF) disalut dengan zink oksida (ZnO) nanorods dilaporkan di sini. Salutan nanorod-nanorod mempertingkatkan gandingan dalam gentian oleh serakan cahaya tetapi juga boleh menyebabkan kebocoran. Penstrukturkan pertumbuhan ke kawasan-kawasan tertentu membolehkan penyerakan daripada ruas yang berbeza di sepanjang gentian yang menyumbang kepada jumlah kuasa terganding. Satu pilin corak ZnO nanorod yang seragam, tumpat dan terjajar dengan tinggi dan yang ditumbuhkan di atas teras POF menggunakan kaedah hidroterma dan kesannya disiasat. ZnO nanorod yang mempunyai masa pertumbuhan 12 jam dan suhu 90 °C telah menyediakan gandingan voltan terbaik. Gandingan sisi diukur dengan faktor sebanyak 2.2 kali lebih baik untuk lapisan pilin corak berbanding dengan lapisan tidak tercorak. Pembentukan berbilang ruas telah juga digunakan untuk penguajaan saluran-saluran pelbagai panjang gelombang di mana jalur-jalur digandingkan secara sisi daripada ruas yang berbeza. Didapati sumber cahaya putih boleh nampak dengan ketara menggandingkan cahaya ke dalam POF berbanding dengan sumber laser infra-merah. Satu model teori tertib pertama diterbitkan untuk menyelakakan kesan corak-corak pilin berskala milimeter (mm) terhadap kecekapan gandingan dengan mengubah lebar dan jarak kawasan bersalut dan tidak bersalut. Lebar lapisan corak pilin ZnO nanorod pada POF teras telah dioptimumkan secara teori untuk gandingan sebelah cahaya dan didapati 5 mm adalah lebar tersebut. Satu pengesahan ujikaji telah dilakukan untuk melengkapkan pengoptimuman dan keputusan ujikaji menunjukkan satu hubungan sekaitan yang baik dengan penyelakuan. Lebar corak pilin ZnO nanorod yang ditumbuhkan atas POF teras besar telah digunakan untuk penderiaan suhu dan wap alkohol pelbagai saluran optik. Laporan pilin corak ZnO nanorod mempamerkan satu tindak balas yang ketara kepada perubahan suhu dari 20 °C hingga 100 °C berdasarkan konsep pemupusan yang
merupakan pengecilan cahaya oleh serakan dan penyerapan apabila ia merentasi ZnO nanorod. Kepekaan diukur yang menunjukan faktor 1.3 kali lebih baik untuk lapiran corak pilin yang bertentangan dengan lapiran tidak bercorak. Mekanisme penderiaan wap alkohol pelbagai saluran optik telah menggunakan perubahan-perubahan di dalam isyarat keluaran disebakan oleh penyerapan wap-wap methanol, etanol dan isopropil. Tiga jalur spektrum terdiri daripada merah (620-750 nm), hijau (495-570 nm) dan biru (450-495 nm) telah digunakan dalam pengukuran ini. Julat nisbi pemodulatan keamanat ditentukan antara 25 hingga 300 ppm. Metanol menunjukkan tindakbals yang kuat berbanding etanol dan isopropil dalam ketiga-tiga saluran spektrum. Dengan mengambil kira nisbi pemodulatan keamanat pengesan alkohol oleh jalur spektrum, saluran hijau menunjukkan nilai nisbi pemodulatan keamanat tertinggi diikuti dengan masing-masing oleh saluran biru dan merah.
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<table>
<thead>
<tr>
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<th>Description</th>
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<tbody>
<tr>
<td>°C</td>
<td>Degree Celsius</td>
</tr>
<tr>
<td>µm</td>
<td>Micrometer</td>
</tr>
<tr>
<td>nm</td>
<td>Nanometer</td>
</tr>
<tr>
<td>cm</td>
<td>Centimeter</td>
</tr>
<tr>
<td>g</td>
<td>Gram</td>
</tr>
<tr>
<td>mM</td>
<td>Mili mole</td>
</tr>
<tr>
<td>C$_2$H$_5$OH</td>
<td>Ethanol</td>
</tr>
<tr>
<td>CH$_3$OH</td>
<td>Methanol</td>
</tr>
<tr>
<td>C$_3$H$_8$O</td>
<td>Isopropanol</td>
</tr>
<tr>
<td>C$<em>6$H$</em>{12}$N$_4$</td>
<td>Hexamethylenetetramine</td>
</tr>
<tr>
<td>HCL</td>
<td>Hydrochloric acid</td>
</tr>
<tr>
<td>CO</td>
<td>Carbon monoxide</td>
</tr>
<tr>
<td>O$_2$</td>
<td>Oxygen</td>
</tr>
<tr>
<td>CO$_2$</td>
<td>Carbondioxide</td>
</tr>
<tr>
<td>H$_2$O</td>
<td>Water</td>
</tr>
<tr>
<td>NaOH</td>
<td>Sodium hydroxide</td>
</tr>
<tr>
<td>ZAH</td>
<td>Zinc acetate hydrate</td>
</tr>
<tr>
<td>Zn(CH$_3$COO)$_2$</td>
<td>Zinc acetate</td>
</tr>
<tr>
<td>Zn(NO$_2$)$_3$</td>
<td>Zinc Nitrate hexahydrate</td>
</tr>
<tr>
<td>Zn(OH)$_2$</td>
<td>Zinc hydroxide</td>
</tr>
<tr>
<td>ZnCl$_2$</td>
<td>Zinc chloride</td>
</tr>
<tr>
<td>ZnO</td>
<td>Zinc oxide</td>
</tr>
<tr>
<td>Zn$^{2+}$</td>
<td>Zinc ions</td>
</tr>
<tr>
<td>O$^{2-}$</td>
<td>Oxygen ions</td>
</tr>
</tbody>
</table>
OH⁻ : Hydroxyl ions

dB/km : Decibels/kilometer

$V_{pp}$ : Peak-to-peak Voltage

$\sigma$ : Beam waist

$r$ : Distance from the center of the beam

$C_{sc}$ : Scattering cross section

$\rho_a$ : Rods density

$\psi$ : Portion of Scattered Light

$\theta_{inc}$ : Incident angle

$\theta_c$ : Critical angle

$\Delta z$ : Width of Segment

$\eta_z$ : Coupling coefficient

$I_p$ : Coupling output of spiral pattern

$I_{up}$ : Coupling output of unpatterned

$n$ : Refractive index

$\Delta I$ : Normalized coupling output

$\phi$ : Azimuthal angle

HMT : Hexamethylenetetramine

MMF : Multimode Fiber

SOF : Silica Optical Fiber

OFSs : Optical Fiber Sensors

POF : Plastic Optical Fiber

AI : Artificial intelligence

PMMA : Polymethyl Methacrylate
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>LED</td>
<td>Light Emitting Diode</td>
</tr>
<tr>
<td>FBG</td>
<td>Fiber Bragg Grating</td>
</tr>
<tr>
<td>DI</td>
<td>Deionized</td>
</tr>
<tr>
<td>DC</td>
<td>Direct Current</td>
</tr>
<tr>
<td>1D</td>
<td>One Dimension</td>
</tr>
<tr>
<td>2D</td>
<td>Two Dimension</td>
</tr>
<tr>
<td>3D</td>
<td>Three Dimension</td>
</tr>
<tr>
<td>ZAH</td>
<td>Zinc Acetate Hydrate</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy-dispersive X-ray</td>
</tr>
<tr>
<td>SPR</td>
<td>Space Plasmon Resonance</td>
</tr>
<tr>
<td>RH</td>
<td>Relative Humidity</td>
</tr>
<tr>
<td>CTOP</td>
<td>Specialty Amorphous Fluorinated Polymer</td>
</tr>
<tr>
<td>DMA</td>
<td>Dynamic Mechanical Analysis</td>
</tr>
<tr>
<td>MPOF</td>
<td>Multimode Plastic Optical Fiber</td>
</tr>
<tr>
<td>OTDR</td>
<td>Optical Time-Domain Reflectometry</td>
</tr>
<tr>
<td>OFDR</td>
<td>Optical Frequency-Domain Reflectometry</td>
</tr>
<tr>
<td>VCO</td>
<td>Voltage-Controlled Oscillator</td>
</tr>
<tr>
<td>RI</td>
<td>Refractive Index</td>
</tr>
<tr>
<td>IR</td>
<td>Infrared</td>
</tr>
<tr>
<td>VZn</td>
<td>Zinc Vacancies</td>
</tr>
<tr>
<td>ca.</td>
<td>Around, about or approximately</td>
</tr>
<tr>
<td>RIM</td>
<td>Relative Intensity Modulation</td>
</tr>
<tr>
<td>GOF</td>
<td>Glass Optical Fiber</td>
</tr>
</tbody>
</table>
LIST OF APPENDICES

Publications and Papers Presented

Patent Filing Reports
CHAPTER 1: INTRODUCTION

1.1 General

Historically, the early research on optical fiber sensors (OFSs) was started in the 70s and related to medical instrument that was such as a fiber-optic endoscope consisting of a bundle of flexible glass fibres able to coherently transmit an image (Edmonson, 1991). Nowadays, various approaches and technologies have been developed to gain attention in sensing applications. Optical sensors using fiber optics definitely provide reliable solutions in many fields since optical fibers can measure physical properties such as strain (Ohno, Naruse, Kihara, & Shimada, 2001), displacement (Rahman, Harun, Yasin, & Ahmad, 2012), temperature (Tyler et al., 2009), pressure (W. Wang, Wu, Tian, Niezrecki, & Wang, 2010), velocity (Weng et al., 2006) and magnetism (Lv, Zhao, Wang, & Wang, 2014). Every year, exploring the potentials of OFSs keep receiving high interest because optical fibers offer well known advantages such as immunity to electrical and magnetic fields, low attenuation, wide transmission bandwidth, small physical size and weight, increased flexibility, analog and digital transmission, electrical insulation, immunity to electromagnetic interference and interception and receiver sensitivity. Beside these properties, FOSs also hold enormous potential for the use in chemical applications due to the high sensitivity and slightly invasive technique (Mescia & Prudenzano, 2013) which is important in monitoring environmental pollution, mainly if FOSs are applied in radiation zone.

This thesis is concerned with the development of a simple and cost effective system based on light scattering from zinc oxide (ZnO) nanorods grown in spiral pattern on plastic optical fiber (POF) for temperature and alcohol vapors sensing applications. The performance of the system is investigated based on the simulation and experimental