UNIVERSITI PUTRA MALAYSIA

# *PALLADIUM-COATED NANOCOMPOSITE ON TAPERED OPTICAL FIBER FOR HYDROGEN SENSING APPLICATIONS*

AL-KHABET MOHAMMED MAJEED MOHAMMED

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By

**AL-KHABET MOHAMMED MAJEED MOHAMMED**

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

**June 2022**

## DEDICATIONS

To

My Father for His Love and Endless Support,

My Mother, Number One for Me

My beloved brothers, The Shoulder to Lean On,

My supervisor,

All my Supervisory Committee,

All of My Friends,

My beloved first and second country Iraq and Malaysia

Without Your Support and Encouragement, My Success Wouldn’t Have Been Possible.

Abstract of 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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**Chairman : Associate Professor Mohd Hanif bin Yaacob, PhD**
**Faculty : Engineering**

Gaseous pollutants such as hydrogen gas ($H_2$) are present in daily human activities and have been studied extensively due to their high explosive and widespread use in many fields. A common $H_2$ gas detector is electrically based. Although these electrical or conductometric sensors attain high sensitivity, they suffer from drawbacks, including poor selectivity, high operating temperature, and susceptibility to electromagnetic interference, which the optical-based sensor can improve. Optical fiber sensors offer advantages over electrical sensors in certain aspects, such as their compact size, the ability to work in harsh environments, and the ability for remote and distributed sensing. However, $H_2$ detection with optical fibers has not been fully explored.

Nanotechnology-enabled chemical sensors have been increasingly used to enhance the sensing performance compared to the conventional sensors toward target analytes owing to their high surface area. The sensing layer based on nanostructures has been identified to work at low temperatures with high sensitivity. Therefore, this research project aims to design and comprehensively analyze optical fiber-based $H_2$ gas sensors by incorporating different nanocomposite coatings as sensing layers. This study uses tapered multimode silica fiber (MMF) sensors as a transducing platform. The tapering process is essential to improve the sensitivity to the environment through the interaction of the evanescent field over the area of the tapered surface area. The tapered area is coated with a sensor layer which is also an essential factor affecting the sensor's performance. The influence of nanostructures' morphology and roughness on the sensing performance were also studied in this Ph.D. research.

The nanostructured materials investigated are graphene oxide (GO), polyaniline (PANI), and molybdenum trioxide ($MoO_3$). These nanomaterials were combined as a nanocomposite sensing layer to enhance the $H_2$ detection. A noble metal, palladium (Pd), was selected as a catalyst to split hydrogen ions. The novel nanocomposites of Pd/GO, Pd/PANI/GO, and Pd/$MoO_3$/PANI were dropped cast on the tapered optical fiber for

sensing analysis. Combining these materials as nanocomposite adds up the functionality to enhance the high surface area to volume ratio to effectively miniaturize and improve the sensing properties of the developed sensors. In this context, nanocomposite materials promote effective $H_2$ gas sensing peculiarity and allow the developed sensors to be operational at low temperatures. Micro-nano characterization techniques such as FESEM, EDX, AFM, and XRD were utilized to obtain detailed structural properties of these nanostructures and fundamentally understand their functions concerning optical sensor performance.

The response of the sensors towards $H_2$ gas was measured at concentrations of 0.125% - 2.00% using optical absorbance change within the wavelength range of 550-850 nm at different temperatures. The sensor performance was evaluated regarding response time, recovery time, sensitivity, repeatability, and stability at different temperatures.

The developed $H_2$ sensors using tapered optical fiber coated with Pd/GO, Pd/PANI/GO, and Pd/$MoO_3$/PANI nanocomposite operated at different temperatures are the first of its kind according to the author's knowledge. The Pd/GO nanocomposite-based sensor demonstrated higher sensitivity of about 33.22/vol% compared to Pd/PANI/GO and Pd/$MoO_3$/PANI nanocomposite, where the sensitivity is about 10.43/vol% and 16.81/vol%, respectively. The response and recovery time of the developed sensors based on Pd/GO, Pd/PANI/GO, and Pd/$MoO_3$/PANI nanocomposite recorded were 48 s, 60 s, and 90 s, and their recovery times were 420 s, 190 s, and 230 s, respectively. Overall, the developed sensor based on Pd/GO nanocomposite showed excellent sensitivity, higher response time, selectivity, and long-term stability compared to Pd/PANI/GO and Pd/$MoO_3$/PANI nanocomposite-based sensors.

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

**NANOKOMPOSIT YANG DISALUT PALLADIUM PADA OPTIK GENTIAN TIRUS UNTUK APLIKASI PENDERIA HIDROGEN**

Oleh

**AL-KHABET MOHAMMED MAJEED MOHAMMED**

**Jun 2022**

**Pengerusi : Profesor Madya Mohd Hanif bin Yaacob, PhD**
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Bahan pencemar gas seperti gas hidrogen ($H_2$) terdapat dalam aktiviti harian manusia dan telah dikaji secara meluas kerana keupayaannya yang mudah meletup dan penggunaan secara meluas dalam banyak bidang. Pengesan gas $H_2$ biasanya berasaskan elektrik. Walaupun penderia elektrik atau konduktometri ini mempunyai kepekaan yang tinggi, mereka mempunyai kelemahan, termasuklah kepilihan yang lemah, suhu operasi yang tinggi dan mudah terdedah kepada gangguan elektromagnetik, yang dapat diperbaiki oleh penderia berasaskan optik. Penderia gentian optik menawarkan kelebihan berbanding penderia elektrik dalam aspek tertentu, seperti saiz yang padat, keupayaan untuk bekerja di persekitaran yang keras dan penderiaan jarak jauh. Walau bagaimanapun, pengesanan $H_2$ dengan gentian optik masih belum diterokai sepenuhnya.

Penderia kimia berteknologi-nano semakin bertambah penggunaannya bagi mempertingkatkan prestasi pengesan berbanding penderia konvensional terhadap analit sasaran kerana luas permukaannya yang tinggi. Lapisan penderia berasaskan nanostruktur telah dikenal pasti berfungsi pada suhu rendah dengan kepekaan tinggi. Oleh itu, projek penyelidikan ini bertujuan untuk mereka bentuk dan menganalisis secara komprehensif penderia gas $H_2$ berasaskan gentian optik dengan menggabungkan salutan nanokomposit yang berbeza sebagai lapisan penderia. Kajian ini menggunakan penderia tirus gentian silika pelbagai mod (MMF) sebagai pelantar transduksi. Proses menirus adalah penting untuk meningkatkan kepekaan terhadap persekitaran melalui interaksi medan evanesen ke atas kawasan permukaan tirus. Kawasan tirus disaluti dengan lapisan penderia yang juga merupakan faktor penting yang mempengaruhi prestasi penderia. Pengaruh morfologi dan kekasaran nanostruktur terhadap prestasi penderia juga dikaji dalam penyelidikan PhD ini.

Bahan berstruktur nano yang disiasat adalah grafin oksida (GO), polianilin (PANI) dan molybdenum trioksida ($MoO_3$). Bahan nano ini telah digabungkan sebagai lapisan penginderaan nanokomposit untuk meningkatkan pengesanan $H_2$. Logam adi, paladium,

telah dipilih sebagai pemangkin untuk memisahkan ion hidrogen. Nanokomposit novel Pd/GO, Pd/PANI/GO dan Pd/$MoO_3$/PANI telah dicicirkan pada gentian optik tirus untuk analisis penderiaan. Kombinasi bahan-bahan ini sebagai nanokomposit menambahkan lagi fungsi serta meningkatkan nisbah luas permukaan kepada isipadu agar menambah baik sifat-sifat penderia yang telah dibangunkan. Dalam konteks ini, bahan nanokomposit mendorong pengesanan gas $H_2$ yang berkesan dan membolehkan penderia yang telah dibangunkan untuk beroperasi pada suhu rendah. Teknik pencirian mikro-nano seperti FESEM, EDX, AFM, dan XRD juga digunakan untuk mendapatkan struktur nano yang terperinci bagi memahami fungsinya secara asas berkaitan dengan prestasi penderia optik.

Tindak balas penderia terhadap gas $H_2$ telah diukur pada kepekatan 0.125 % - 2.00 % menggunakan perubahan serapan optik dalam julat panjang gelombang 550-850 nm pada suhu yang berbeza. Prestasi penderia telah dinilai dari segi masa tindak balas, masa pemulihan, kepekaan, kebolehulangan dan kestabilan jangka panjang pada suhu yang berbeza.

Penderia $H_2$ yang telah dibangunkan menggunakan gentian optik tirus yang disalut nanokomposit Pd/GO, Pd/PANI/GO dan Pd/$MoO_3$/PANI telah beroperasi pada suhu yang berbeza adalah yang pertama seumpamanya mengikut pengetahuan penulis. Penderia berasaskan nanokomposit Pd/GO menunjukkan kepekaan yang lebih tinggi sekitar 33.22/ vol% berbanding dengan nanokomposit Pd/PANI/GO dan Pd/$MoO_3$/PANI di mana kepekaan masing-masing adalah sekitar 10.43 /vol% dan 16.81 /vol%. Masa tindak balas dan pemulihan penderia yang telah dibangunkan berasaskan nanokomposit Pd/GO, Pd/PANI/GO dan Pd/$MoO_3$/PANI yang telah direkodkan adalah masing-masing 48 s, 60 s dan 90 s, dan masa pemulihannya adalah masing-masing 420 s, 190 s dan 230s. Secara keseluruhan, penderia yang dibangunkan berasaskan nanokomposit Pd/GO menunjukkan kepekaan yang sangat baik, masa tindak balas yang lebih tinggi dengan kepilihan yang lebih tinggi dan kestabilan jangka panjang berbanding dengan penderia berasaskan nanokomposit Pd/PANI/GO dan Pd/$MoO_3$/PANI.

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finally,

My sincere thanks to all these gentlemen!

Mohammed Majeed Mohammed Al-Khabet

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Date: 8 December 2022

**Declaration by Members of Supervisory Committee**

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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Signature: ______________________________
Name of Member of Supervisory Committee: Associate Professor Ir. Dr. Norhana binti Arsad

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| | |
|---|---|
| 1D | One dimensional |
| 2D | Two dimensional |
| AFM | Atomic force microscopy |
| APS | Ammonium persulfate |
| $CH_4$ | Methane gas |
| CRDS | Cavity Ring-Down Spectroscopy |
| DEP | Dielectrophoretic |
| EDX | X-ray energy dispersion |
| EMI | Electromagnetic Interference |
| EP | Electrode potential |
| EW | Evanescent wave |
| FBG | Fiber Bragg grating |
| FCC | Face-cantered cubic |
| FESEM | Field emission scanning electron microscopy |
| FOS | Fiber Optics Sensor |
| GO | Graphene oxide |
| $H_2$ | Hydrogen gas |
| $H_2SO_4$ | Sulfuric acid |
| HF | Hydrochloric acid |
| ICDD | International Center for Diffraction Data |
| IR | Infrared |
| $K_2PdCl_6$ | Potassium hexachlorophylate |
| $KMnO_4$ | Potassium permanganate |

| | |
|---|---|
| MMF | Multimodal mode fiber |
| $MoO_3$ | Molybdenum trioxide |
| MOSFET | Membrane-Oxide-Semiconductor Field-Effect Transistors |
| $NaBH_4$ | Sodium borohydride |
| NASA | National Aeronautics and Space Administration |
| $NH_3$ | Ammonia gas |
| $NO_2$ | Nitrogen dioxide |
| PANI | Polyaniline |
| PAS | Photoacoustic Spectroscopy |
| PCF | Photonic crystal fiber |
| Pd | Palladium |
| $PdCl_2$ | Palladium chloride |
| PdHx | Palladium hydride |
| POF | Plastic optical fibers |
| RI | Refractive Index |
| SAW | Surface acoustic wave |
| $SiO_2$ | silicon dioxide |
| $SO_2$ | Sulfur dioxide |
| SOF | Silica Optical Fibers |
| SPR | Surface plasmon resonance |
| $TiO_2$ | Titanium dioxide |
| TIR | Total Internal Reflection |
| TR | Temperature resistance |
| USB | Universal serial bus |

| | |
|---|---|
| UV | ultra-violet |
| VGPS | Vytran Glass Processing System |
| VIS | Visible |
| WFV | Work function voltage |
| XRD | X-ray diffraction |
| ZnO | Zinc oxide |

# CHAPTER 1

# INTRODUCTION

This chapter provides an overview of the Ph.D. research. Section 1.1 provides a research background and motivation on hydrogen gas and its sensing technologies. Section 1.2 presents the recent advances in optical fiber sensors integrated with nanostructured materials. Section 1.3 discusses the problem statement related to hydrogen sensing using optical fiber. Section 1.4 outlines the research objectives and questions related to the Ph.D. project. Finally, sections 1.5 and 1.6 present the research's scope and the thesis's organization.

## 1.1 Research Background and Motivations

Hydrogen ($H_2$), owing to its high efficiency as a fuel, abundance, non-polluting character, and sustainability, is one of the possible solutions to the impending energy crisis [1], [2]. It is also a dependable gas for faulty power transformers diagnostics [3], [4]. $H_2$ has also been used in other sectors such as aerospace engineering, metallurgical refineries, petroleum explorations, chemical processing, cryogenic cooling, and many more [5]–[10]. However, its high diffusion coefficient (0.16 $cm^2/s$ in the air), low ignition energy (0.018 mJ), wide explosion concentration range (4% -75%), and high heat of combustion (285.8 kJ/mol) make it a highly explosive gas and potentially dangerous for us, transport and storage [11]–[13].

Currently, many different technologies are being used or under development for the detection of hydrogen, including semiconductor sensors, electrochemical, thermal sensors, and mass spectroscopy [14] [15]. Unfortunately, these sensing methods have several disadvantages, such as large size, high cost, dependence on the presence of oxygen, and the potential to create electrical sparks that would be dangerous in explosive environments. There are four main hydrogen sensor types: chemical resistance, surface acoustic waves, optical fiber sensor, and microelectronic sensor [16]. However, these sensors are limited by several drawbacks, such as low selectivity, high power consumption, and electromagnetic interference (EMI), which limit their use in practical applications. Furthermore, all of the above sensors require a high operating temperature (> 300 °C) and many moving mechanical elements, which leads to implementation problems for remote sensing applications [17]. Thus, to overcome these drawbacks, optical fiber has been examined as an alternative to conventional chemical sensors due to their many advantages. Optical fiber sensors are easily integrated into optical networks and communication systems with additional distributed remote sensing functions [18]. The introduction of chemical-based optical fiber sensors has received much attention recently due to their small size, immunity to electromagnetic interference, and ability to work in harsh environments [19]. Since then, optical fiber-based chemical sensors have been adopted in various applications, and enormous efforts have been made to improve their performance.

## 1.2 Nanostructure Coated Optical Fiber Sensor

Optical fiber sensors have gained huge popularity and acceptance in the market in recent years due to the number of advantages that their electrical counterparts have. These advantages include miniaturization, flexibility, and immunity to electromagnetic interference, as well as its ability to operate in harsh environments, such as high temperatures or a chemically reactive environment [20]. Optical fibers are also inert, passive, and electrically non-conductive. Therefore, optical fibers are safe to use near flammable or explosive environments, as in the case of the oil and gas industry [21].

Recent advances in sensing technology include the development of optical fiber sensors integrated with nanomaterials. Nanostructures are materials with at least one dimension in the nanoscale range, less than 100 nm [22]. These materials are particularly suitable for chemical and biological detection applications because many reactions occur at the nanoscale. When materials are reduced to nanoscale dimensions, they reveal new and unique properties, such as a high surface-to-volume ratio, high heat capacity, mechanical strength, and changes in magnetic behavior. The rapid development of nanoparticle synthesis and nanofabrication methods leads to the development of many new nanomaterials with unique physical and chemical properties [23]. Among those interesting properties are the material's optical characteristics, including reflectivity, absorbance, and fluorescence [24].

Optical fiber sensors with nanomaterials enhancements provide an exciting new way for chemical sensing applications. Incorporation of nanomaterials thin film with optical fiber significantly improves the chemical sensing performance [25]. Optical techniques commonly used to measure response in chemical detection are reflectance, absorbance, fluorescence, and surface plasmon resonance which result from the interaction between the nanomaterials coated optical sensor with various chemical molecules [26].

## 1.3 Problem Statement

Most chemical sensors are electrical-based systems such as conductometric, quartz crystal microbalance, and Schottky diode, to name a few. The main reasons for the extensive use of electrical sensors are their high sensitivity and low cost. However, these sensors suffer from poor selectivity and limited application in a volatile environment, particularly where a high risk of explosion or exposure to electromagnetic ignition is expected. Hazardous chemicals, such as $H_2$ gas, are flammable and explosive. The limitations of electrical-based sensors in detecting these chemicals can be addressed using optical transducers, such as optical fiber.

The development of optical fiber sensors is relatively less established than electrical sensors. Nevertheless, the reduced cost of optical components, driven by the large commercial telecommunication and optoelectronic markets, creates an opportunity to implement the technology for $H_2$ sensing applications. The optical signal properties have many advantages compared to the electrical signal and thus offer the vast opportunity to the researchers to explore its potential in the $H_2$ sensing applications. Some of the unique properties of optical signals, such as immunity to EMI and resistance to corrosive, reactive, and flammable environments, make optical fiber sensors a promising candidate for $H_2$ sensing applications.

Last decades, most of the sensing layers deployed in chemical detection are based on thick films (approximately more than 10 μm). Nevertheless, the advancement of nanotechnology provides an opportunity to integrate sensing materials at the nanoscale with transducing platforms. Nanostructured materials have numerous advantages, including a large surface-to-volume ratio, high specific surface area, and several surface active sites [27][28]. Recent studies have identified that using nanostructured material as an active sensing layer can improve the chemical sensing performance concerning sensitivity, selectivity, and response time compared with thick film sensing layers.

Most of the reported optical fiber sensors for $H_2$ sensing are based on special optical fibers such as plastic optical fiber (POF), photonic crystal fiber (PCF), and fiber Bragg grating (FBG). These sensings involve expensive and complicated fabrication techniques. As a result, the author believes that by employing optical fiber sensors in simpler and cost-effective fabrication in volatile environments, the safety risks associated with the leakage of $H_2$ can be significantly reduced.

## 1.4 Research Objectives and Questions

This thesis focuses on developing tapered optical fiber sensors coated with nanomaterials for $H_2$ sensing applications. In order to achieve this, the following objectives are outlined:

1. To design and fabricate $H_2$ gas sensors based on nanomaterials coated on tapered optical fiber via drop-casting technique.

2. To synthesize and characterize the nanomaterials suitable for the $H_2$ sensing layer.

3. To investigate and evaluate the optical sensing performance of the nanomaterials toward $H_2$ gas.

4. To propose and explain the chemical sensing mechanism of the developed optical fiber sensors.

To achieve these goals, the following research questions were identified:

1. What sensitive nanomaterials change their optical properties when interacting with $H_2$ gas?

2. How can these materials be synthesized and coated onto the tapered optical fiber?

3. How is the sensing performance of tapered optical fiber sensors coated with different nanostructured sensor layers?

4. How to understand the nature of the interaction between the target chemical and the sensor layer?

## 1.5 Research Scope and Limitation

This research project covers the development of tapered optical fiber for $H_2$ sensing based on nanomaterials that lead to optical devices. The dimension profile is fixed to waist diameter to 20 μm with a fixed length of 10 mm and an up / down taper of 5 mm. This profile is well-established and adequate to provide high sensitivity suitable for $H_2$ gas sensing and easy to handle. The optimum operating temperature for $H_2$ sensing was tested, and the largest response can be obtained. Various parameters such as sensitivity, repeatability, and stability of the fabricated sensors were determined, and selectivity toward other gases was also measured. Optimization of the developed optical fiber sensors could not be improved in terms of humidity effect due to the limited time frame for this work. Such analysis will provide these sensors' overall performance for harsh environments. The project scope can best be explained using the tree diagram in Figure 1.1. The solid lines represent the direction in this thesis to achieve the goal and objectives of the work, while the dotted lines indicate other research areas outside the scope of this work. The highlighted squares represent the elements deployed to achieve the research objectives proposed for developing an optical fiber for $H_2$ sensing.

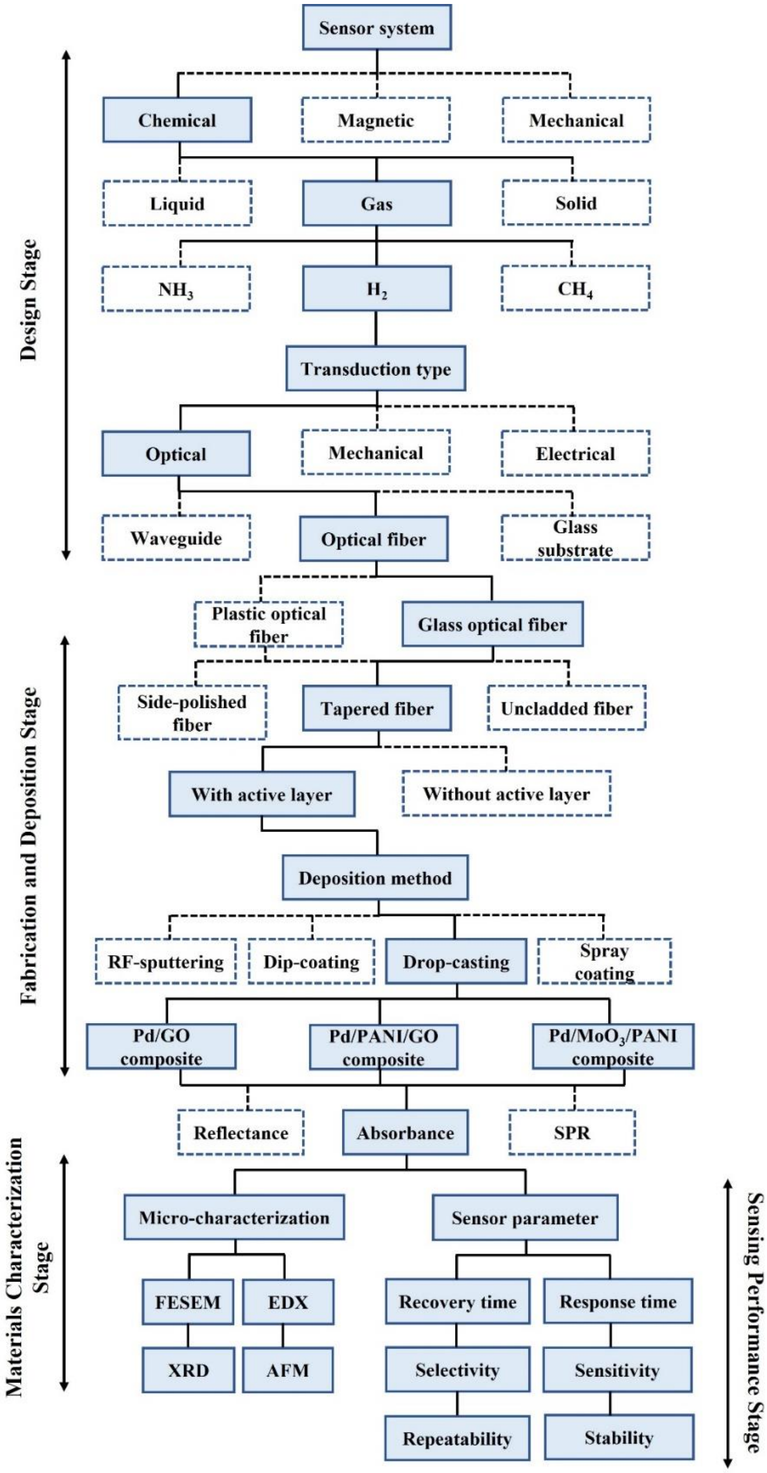


**Figure 1.1: Research Scope**

## 1.6 Thesis Organization

The thesis consists of six chapters. Chapter 1 offers an overview of the research area focusing on the problems that motivated this work. In addition, this chapter clarifies the problem's facts and the investigation's goals and objectives. Chapter 2 comprehensively reviews the types, characteristics, and principles of optical fiber measurement. This chapter also critically reviews optic fiber sensors used in $H_2$ applications. The nanostructured materials used in this doctoral work are included, and their properties and applications are discussed. Chapter 3 provides a complete description of the tapered optical fiber sensor design and fabrication. The synthesis and deposition of nanomaterials are presented and discussed, followed by a gas testing setup for the developed optical fiber sensors. Chapter 4 presents the micro characterization results of the $H_2$-sensitive nanostructures. Chapter 5 discusses gas sensing results from the tapered optical fiber sensor performance combined with the nanomaterials. The investigated optical sensing results include sensitivity, selectivity, response, and recovery times. The optical $H_2$ sensing mechanisms of the developed sensors are also explained in this chapter. Finally, chapter 6 concludes the thesis and provides a list of the contributions of this work. Potential ideas are also suggested to be pursued as future research work.

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