UNIVERSITI PUTRA MALAYSIA

OPTICAL CODE DIVISION MULTIPLE ACCESS-BASED AMMONIA GAS SENSOR NETWORK USING MODIFIED SINGLE MODE FIBER COATED WITH POLYANILINE/GRAPHITE NANOFIBER

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By

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Optical fiber sensor network has received an increasing attention in recent years. This is due to the fact that it has proven to be a crucial platform for monitoring a wide range of parameters in many fields. Optical fiber sensor network which consolidates optical fiber sensors is still in infancy stage especially its applications for chemicals or gas sensing. Some unique properties of optical signal such as immunity to the EMI, resistance to the corrosive and flammable environments make optical fiber a promising candidate for gas sensing applications. Important influence of the sensing layer morphology towards gas sensing performance leads to the deployment of nanomaterials. Nanomaterials based optical fiber sensors are expected to produce highly sensitive optical gas sensors. The sensors can be integrated as a part of optical fiber sensor network for remote and distributed real time in-situ gas monitoring system. One of the efficient ways to manage the multiple sensing nodes in the optical fiber sensor network is by deploying spectral amplitude coding based optical code division multiple access (SAC-OCDMA). SAC-OCDMA is low cost technique and has the ability to suppress the multiple access noise (MAI).

In this project, single mode fibers (SMF) were modified and coated with polyaniline (PANI) nanofiber and PANI/graphite nanofiber (GNF) nanocomposite to produce highly sensitive optical ammonia (NH_3) sensors. GNF was reported to have a unique structure where it has virtually open edges and large interlayer spacing, which also believed to be useful for different applications such as supercapacitor and sensing applications. These sensors were tested towards NH_3 in the visible and C-band wavelengths ranges which is not yet explored for optical NH_3 sensing applications and enables the integration of the sensors with the existing optical fiber communication systems such as fiber to the home (FTTH). NH_3 is selected for the project because it

is highly dangerous gas and widely used for industrial applications. A novel modified SMF that underwent both etching and tapering processes was developed to produce fiber with rough surfaces and reduced cladding structures. Three NH_3 etched-tapered SMF sensors coated with PANI/GNF nanocomposite were multiplexed using SAC-OCDMA technique to establish a star topology optical fiber sensor network.

The SAC-OCDMA technique deployed in the optical fiber sensor network for NH_3 sensing is based on Khazani Syed (KS) code. KS code is preferred because it reduces the number of FBG filter and thus, reduces the cost and complexity of the developed system.

At device level, the sensor performance was evaluated in terms of response and recovery times, low limit of detection (LOD), sensitivity and repeatability. At the optical fiber sensor network level, the optical signal to noise ratio (OSNR) was investigated for the developed NH_3 sensing network.

The proposed etched-tapered sensors coated with PANI nanofiber outweight the performance of tapered SMF and etched SMF in terms of sensitivity and response time. The SMF sensors coated with PANI/PGN nanocomposite exhibited superior response as compared to the sensors coated with PANI thin films only towards NH_3 in the visible and C-band wavelengths ranges. The response time and sensitivity of SMF sensors coated with PANI/PGN nanocomposite towards NH_3 was 58 s, 49 s, 300 and 306.8, respectively in the visible and C-band wavelengths ranges.

LOD was found to be approximately 0.04% (400 ppm) at room temperature. These sensors were integrated with the developed optical fiber sensor network and investigated for real time remote sensing with 3 km SMF link using erbium doped fiber amplifier (EDFA). The measured OSNR for SAC-OCDMA based optical fiber sensor network was 20.1 dB when sensors were implied in the network. For remote monitoring with 3 km link only and including the EDFA with 20 dB gain, the OSNR was 19.6 and 31.75 dB, respectively. The use of EDFA improved the OSNR significantly.

In summary, different modified SMF sensors coated with nanostructured thin films were successfully developed and investigated towards NH_3 gas. Strong optical sensing performance showed by the novel fiber sensors indicate their potential to be multiplexed in optical sensor networks for remote as well as distributed NH_3 detection.

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

RANGKAIAN PENDERIA GAS BERASAKAN CAPAIAN BERBILANG PEMBAHAGI KOD OPTIK MENGGUNAKAN GENTIAN MOD TERUBAH TUNGGAL BERSALUT GENTIAN NANO POLYANILINE/GRAFIT

Oleh

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Kebelakangan ini, rangkaian penderiaan gentian optik telah menarik perhatian daripada sektor industri dan penyelidikan. Keadaan ini terbukti dan menjadi platform penting untuk memantau pelbagai parameter dalam banyak bidang. Namun, rangkaian penderiaan gentian optik yang menggabungkan penderiaan gentian optik masih berada pada peringkat awal terutamanya dalam aplikasi yang melibatkan bahan kimia atau penginderaan gas. Isyarat optik mempunyai ciri-ciri yang unik seperti kalis EMI, tahan reput dan persekitaran yang mudah terbakar menjadikan gentian optik ini sesuai untuk diaplikasikan dalam penderiaan gas. Morfologi lapisan penderiaan sangat penting dalam mempengaruhi prestasi penderiaan gas dan ini mengalakkan penggunaan bahan nano untuk tujuan ini. Penderiaan menggunakan bahan nano ke atas gentian optik dijangka akan menghasilkan penderiaan gas yang sangat sensitif. Penderia boleh diintegrasikan sebagai sebahagian daripada rangkaian penderiaan gentian optik untuk sistem pemantauan gas berjarak jauh dan dalam sistem pemantauan agihan penderiaan gas secara langsung. Salah satu cara yang berkesan untuk menguruskan pelbagai nod penderiaan dalam rangkaian penderiaan gentian optik adalah dengan menggunakan teknik SAC-OCDMA. Kaedah ini menjimatkan dan mempunyai kelebihan dalam menindas pelbagai laluan isyarat bunyi (MAI).

Dalam projek ini, gentian mod tunggal (SMF) telah diubahsuai dan dilapis dengan bahan nano PANI dan PANI/Graphite (GNF) nanokomposit untuk menghasilkan penderia optik yang sensitif terhadap ammonia (NH_3). GNF dilaporkan mempunyai struktur yang unik di mana bahan ini mempunyai jarak lapisan yang besar dan hujung terbuka dan ini dikatakan berguna untuk pelbagai aplikasi seperti superkapasitor dan penderiaan. Penderiaan ini diuji terhadap NH_3 dalam gelombang cahaya boleh-lihat dan dalam julat panjang gelombang C yang masih belum diterokai untuk NH_3

penderiaan optikal agar integrasi penderiaan dengan komunikasi gentian optik sediaada seperti FTTH dibolehkan. NH_3 dipilih dalam projek ini disebabkan gas ini amat bahaya namun kerap digunakan dalam sektor perindustrian. Untuk pertama kalinya, gentian optik yang telah diubahsuai dengan punar dan menirus gentian optik SMF bagi menghasilkan permukaan kasar dan struktur pelapis yang telah dikurangkan. Tiga penderiaan yang telah disediakan dengan punar-tirus SMF yang disalut dengan PANI/GNF nanokomposit, dipelbagai-rangkaikan menggunakan teknik SAC-OCDMA untuk menghasilkan topologi berbentuk bintang dalam rangkaian penderiaan gentian optic. Teknik SAC-OCDMA yang digunakan dalam rangkaian pengesan gentian optik untuk mengesan NH_3 adalah berdasarkan kepada kod Khazani Syed (KS). Kod KS lebih dipilih kerana ia mengurangkan jumlah penapis FBG sekaligus mengurangkan kos dan kerumitan sistem yang dibangunkan.

Dari segi peranti, prestasi pengesan ini dinilai mengikut masa yang diambil untuk bertindak balas dan pemulihan, had pengesanan terendah (LOD), kepekaan dan kebolehulangan. Di peringkat rangkaian pengesan gentian optik, isyarat optikal kepada nisbah bunyi (OSNR) telah disiasat untuk rangkaian pengesanan gas NH_3.

Cadangan pengesan punar-tirus yang disalut dengan ketebalan PANI nanofiber menunjukkan banyak kelebian dalam prestasi mengesan jika dibandingkan dengan SMF tirus dan SMF punar dari segi sensitiviti dan masa tindak balas. Pengesan SMF yang disalut dengan nanokomposit PANI/PGN menunjukkan tindak balas yang lebih baik berbanding dengan sensor yang dilapisi dengan filem tipis PANI ke atas gas NH_3 dalam julat panjang gelombang (600 - 750 nm) dan C-julat (1535 - 1565 nm). Masa tindak balas dan kepekaan pengesan SMF yang disalut dengan nanokomposit PANI / PGN terhadap NH_3 masing-masing adalah 58 s, 49 s, 300 s and 306.8, dalam jarak panjang gelombang C dan boleh-lihat.

LOD diperoleh adalah lebih kurang 0.04% (400 ppm) pada suhu bilik. Pengesan ini diselidiki untuk pengesanan jarak jauh dengan SMF sepanjang 3 km dengan menggunakan erbium doped fiber amplifier (EDFA). OSNR yang diukur untuk rangkaian pengesan gentian optik berasaskan SAC-OCDMA adalah sebanyak 20.1dB. Bagi penderiaan jarak jauh 3 km dan melibatkan EDFA dengan 20 dB faedah, OSNR yg dikira adalah sebanyak 19.6 dan 31.75 dB. Penggunaan EDFA telah memperbaiki OSNR secara jelas.

Ringkasnya, pelbagai pengesan SMF yang diubah suai dengan menyalut lapisan nipis bersaiza nano berjaya dibangunkan dan diuji dengan gas NH_3. Prestasi tinggi yang dihasilkan oleh pengesan optik yang novel menunjukkan potensi mereka untuk dikombinasikan dalam rangkaian pengesan optik jarak jauh dengan gas NH_3.

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

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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) were adhered to.

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
ATR	Attenuated total internal reflection
BCP	Bromocresol purple
CDMA	Code division multiple access
CSA	Camphorsulfonic acid
CH_4	Methane
CPR	Chlorophenol red
DWDM	Dense wavelength division multiplex
EB	Emeraldine base
EDFA	Erbium doped fiber amplifier
ES	Emeraldine salt
FBG	Fiber Bragg Grating
FMCW	Frequency modulated carrier wave
GNF	Graphite nanofiber
GNF-H	Graphite nanofiber herringbone type
GNF-P	Graphite nanofiber platelet type
GNF-R	Graphite nanofiber ribbon-type
He-Ne	Helium-neon
HCl	Hydrochloric acid
HF	Hydrofluoric acid
HSPON	Hybrid sensing passive optical network
ITO	Indium tin oxide
IOT	Internet of things
IR	Infrared

KS	Khazani Syed
LEB	Leucoemeraldine base
LED	Light-emitting diodes
LOD	Limit of detection
M	Number of mapping
MAI	Multiple access interference
MFH	Modified frequency-hopping code
Mid-IR	Mid-Infrared
MMF	Multimode Fiber
MOF	Metal organic frame network
MQC	Modified congruence code
NH_3	Ammonia
NNI	Nanotechnology Initiative
OCDMA	Optical code division multiple access
ONU	Optical network unit
OOC	Optical orthogonal code
OS-CDMA	Optical spectrum CDMA
OSA	Optical spectrum analyzer
OSU	Optical sensing units
PANI	Polyaniline
PANI-CSA	Camphorsulfonic acid-doped polyaniline
PANI-EB	Emeraldine base polyaniline
PANI-ES	Emeraldine salt polyaniline
PANI/GNF	Polyaniline/graphite nanofiber
PIIN	Phase induced intensity noise

PMMA	Poly (methyl methacrylate)
PMMA/CPR	Poly (methyl methacrylate)/chlorophenol red
PON	Passive optical network
ppb	Part per billion
pm	Picometer
ppm	Part per million
PRBS	Pseudorandom bit sequence
SAC-CDMA	Spectral amplitude coding Optical code division multiple access
SEM	Scanning electron microscope
SDM	Space division multiplexing
SNR	Signal-to-Noise Ratio
SOA	Semiconductor optical amplifier
SDD	Spectral direct decoding
T-WDM	Hybrid time and wavelength division multiplexing
TDM	Time division multiplexing
TSL	Total system loss
UV	Ultraviolet
UV-Vi	Ultraviolet-visible
WDM	Wavelength division multiplexing
WO_3	Tungsten trioxide

CHAPTER 1

INTRODUCTION

1.1 Motivations

In the past decades, optical fiber has engaged vital role in the development of optical communication systems, as well as in the development of telecommunication industry. Intensive studies [1] have carried out using optical fiber not limited to communication networks only but they have expanded to consolidate the optical fiber sensor networks and development of optical fiber sensors. There is a drastic increase in the sensor research, particularly its fabrication and applications. Particularly, the development of gas sensors resulted in a revolution similar to the trend experience by computers in the 1980s. The first decade of 21st century has been called as sensor decade [2]. Tremendous advances have been made in sensor technology and many more are yet to come.

Currently, the focus of the research in the sensing layer materials is nanoscale materials. Nanomaterials can be integrated with the optical sensors. Nanomaterials can be partitioned into three types categories, specifically, semiconducting metal oxides (inorganic), conducting polymers (natural) and composite materials [3]. In this sense, the extrinsic optical fiber sensor is deployed. Normally, the fiber guides the light to and from the chemical sensing layer where the light experienced a modulation due to the interaction between the chemical sensing layer and the chemical analyte [4].

The use of optical fibers in sensing applications like physical, chemical and biochemical sensing has made much progress since its developments began in the 1960s. The conventional gas sensors are based on electrical signal. Although electrical-based sensors are well-established technology, these sensors suffer from some restrictions, which can be avoided by using optical fiber sensors. Since optical fibers are dielectric medium, has no electrical signal conductivity, they can stand those harsh environments. It is immune to electromagnetic interference and can stands high temperature up to 1200ºC before it start to soften [5]. Moreover, the fiber is inexpensive and small size. In addition to abovementioned merits, these sensors can also be utilized for refractive index measurement in which only a small volume of sample is required [6]. Therefore, optical fiber sensors have been used to monitor a wide range of parameters such as pH, humidity, concentrations of gases, voltage, temperature, pressure, vibration, specialty chemicals, acoustic emission and fracture [7]. The optical fiber sensors are considered a promising candidate for gas sensing applications.

It is necessary to get a continuous and reliable monitoring system to assist in minimizing the risk of human exposure to the hazardous gases by developing simple, fast and safe transducers [8]. Furthermore, optical fiber sensors have been developed and gained popularity as practical and highly sensitive devices towards chemicals with low concentrations. The optical fiber sensors provide possibility of real time monitoring distributed and remote sensing for wide area coverage [6, 9]. This is due to their established applications in the long distance telecommunication networks [10].

Applying optical fiber sensors for gas sensing applications has opened up new possibilities of in-situ monitoring on various types of gases at remote or hard-to-reach areas. Remote monitoring enables real-time and continuous monitoring of certain gas species is in huge demand in process control, automotive, medical and many more. Remote monitoring whereby a group of optical fiber gas sensors spreads over wide area away from the control center necessitates the use of a multiplexing technique and optical fiber sensor network. Optical fiber sensor networks, generally, can be defined as a group of two or more optical fiber multiplexed sensors which are deployed either directly inside the element to be assessed or very close to it. This requires a scheme to provide a definite sensor addressing (or multiplexing) and interrogation (or demodulation). The most fundamental motivation for multiplexing optical fiber sensors is the cost [11]. The cost of a single channel optical fiber sensor is relatively high. Fortunately, aggregation of the sensors results in cost reduction, given that it would be possible to share either the source of light, system of detection, or, preferably both. Thus, the main goal of most optical fiber sensors networks, is to connect or multiplex a number of sensors to a single detection unit. These sensors can be addressed either simultaneously or sequentially by using optical switches [12]. The main motivation to specifically develop optical fiber gas sensors and related networks is due to the health and safety concerns that have arisen with the increase of hazardous gases in the environment due to the pollutions over wide areas.

Different sensing techniques are used for the optical fiber sensors to detect gas that includes interferometric based [8], microbending [13], grating [14], refractive index and evanescent wave [15, 16]. In recent years, evanescent field based optical fiber sensors have become increasingly popular for remote and distributed sensing applications. The main advantage of these sensors is that one can monitor the parameter of interest (e.g. gas concentration) in real time and in situ [17]. Evanescent wave sensor incorporates some modification on the sensing area of the optical fiber.

One of the common used gas in industry is ammonia (NH_3). NH_3 is a severe respiratory tract irritant. Numerous cases of fatal ammonia exposure have been reported, but actual exposure levels have not been well documented. Severe short-term exposures to NH_3 leads to long-term respiratory system and lung disorders. People repeatedly exposed to ammonia may develop a tolerance (or acclimatization) to the irritating effects after a few weeks. These safety concerns emphasize the importance of the ammonia sensor and its huge potential in the future. Due to above issues, it is necessary to get a continuous and reliable remote supervision and monitoring system of NH_3 to assist in minimizing the risk of human exposure to the gases [18, 19].

Recently, use of the modified optical fiber sensors have aroused much interest due to significant features such as robustness, strong evanescent field, compactness and simple fabrication processes. Modified optical fiber sensors have been studied for measuring physical parameters such as temperature [20], humidity [21], strain [22], refractive index [23] as well as for detecting chemicals species [24] and biosensors [25]. Great potentials of modified optical fiber sensors are now identified by the research community particularly the optical fiber gas sensors. Integration of nanomaterials and optical fiber sensors for volatile environments can be a strong alternative to electrical sensors in near future due to minimum risk at general emergencies such as ammonia leakage. Combining the modified optical fiber with highly sensitive nanomaterials in ammonia sensing applications is an interesting research field to be explored with significant novelty.

1.2 Problem Statement

An important advantage of optical sensors, specifically optical fiber sensors, is their ability to be used for remote sensing to cover wide areas. This is due to their established applications in the long-distance telecommunication networks. However, the use of gas optical fiber sensor toward gas monitoring via a communication network is still in its infancy stage. There is a vast opportunity in this research area to establish gas sensor network using modified optical fiber sensor coated with nanostructured thin films. There is also an increasing demand to establish the remote monitoring by deploying the existing optical communication network infrastructures. This is highly impact to simplify the network system design and reduce the cost of the chemical remote monitoring.

The common NH_3 sensors are based on electrical signal. Even though it is simple and low cost; the electrical based sensor has poor selectivity by responding to other gases. Furthermore, the sensor is susceptible towards electrical noise such as electromagnetic interference (EMI) and its application is localized. In the volatile environment such as oil and gas plants, the electrical based sensors are not suitable due to possibility of ignition from the signal. There is a crucial demand to find a substitute sensor for detection and cautionary to avoid crises due to NH_3 leakage or drawbacks of the electrical sensors. The optical fiber sensor is an excellent candidate to avoid these drawbacks introduced by the conventional sensors.

The existing NH_3 optical fiber sensors are mostly based on multimode optical fiber (MMF) that operating in the visible wavelengths range. The MMF based sensors are less sensitive than the SMF based sensors that not completely explored for NH_3 sensing. Deploying optical fiber sensors, particularly, highly sensitive modified SMF optical fiber sensors, coated with nanomaterials nanostructured thin film towards NH_3 can help to prevent disasters due to leakage of NH_3. The SMF based sensors can be operated in the C-band wavelengths and hence can be integrated easily with the established optical fiber communication network such fiber to the home.

Modified optical fiber sensors coated with nanostructured thin films have been developed and gained popularity as practical devices towards chemicals such as gases with low concentrations. It is expected that highly sensitive and fast response sensors will be realized by employing these configurations. However, new nanomaterials developed such as polyaniline (PANI), PANI/graphite nanofiber (GNF) nanocomposite and are yet to be fully explored as a sensing layer towards NH_3 [26]. The light weight, high conductivity and low cost leads to the use of PANI. PANI is attractive to be used as a sensing layer because it can rapidly switch between the emeraldine base (EB) and protonated emeraldine salt (ES) forms as it is exposed to certain analytes. GNF has a unique structure where it has virtually open edges and large interlayer spacing, which also believed to be useful for different applications such as supercapacitor and sensing applications [27]. Use of GNF is mainly to improve the sensitivity and selectivity of the sensors towards NH_3.

The SMF sensors can be incorporated in remote gas optical fiber sensor networks to cover wide area in the C-band wavelengths range. Thus, it is easily to be integrated with the existing optical communication networks operate in the C-band range. Techniques like wavelength division multiplexing (WDM), the time division multiplexing (TDM) and additionally optical code division multiple access (OCDMA) have been suggested for multiplexing the yield of optical fiber transducers. The low scanning speed limitation in TDM which makes it not suitable for real time remote measurement as well as the spectral width separation and high cost of multiple wavelength light source in WDM have been considered as hindrances to their applications. Code Division Multiplexing (CDM) known for asynchronous transmission capability and information security has challenges with Multiple Access Interference (MAI). OCDMA is a system that is used to combine multiple optical signals from different sources using different optical codes. The system is commonly deployed in the telecommunication field for long distance and high capacity transmission. However, the SAC/OCDMA system is yet to be widely utilized in optical fiber sensor networks.

1.3 Aims and Objectives

The aim of this research is to design and demonstrate an SMF optical fiber gas sensor network based on OCDMA for remote monitoring application. The gas under testing is NH_3 due to its high severity and deployment in the industry. The objectives to achieve this research project are as follows:

- to design, fabricate and characterize modified SMF transducing platforms, that are etched, tapered and etched-tapered SMF platforms.
- to synthesize, deposit, characterize and evaluate gas sensing characteristics of the nanomaterials (PANI nanofiber and PANI/graphite nanofiber (GNF) nanocomposite) as a sensing layer onto developed modified SMF transducing platforms towards different concentrations of NH_3 gas within visible and C-band wavelength ranges.
- to design and develop optical gas sensor network deploying modified SMF sensor and Khazani Syed (KS) code based SAC/OCDMA technique.
- to investigate and evaluate the modified SMF sensor performances for real time remote and distributed monitoring systems using SAC/OCDMA gas sensor network deploying EDFA.

1.4 Limitations and Boundary of the Reseach

Figure 1.1 represents the scope of this PhD research work. This project implies mainly two parts, the NH_3 sensor characterization and optical fiber gas sensor network development. The main boundary related to sensing characterization is the parameters used throughout thesis are well optimized. This for the parameters of the etching and tapering of the SMF. Some of preliminary and raw results are included in Appendix C.

The dynamic response of modified SMF sensors were investigated towards NH_3. No investigations were done regarding the effects of environment and aging on the modified coated fiber i.e. stability over time. Moreover, the EDFA used in the optical fiber gas sensor network was only to amplify the optical signal with different gains. The other properties related to the use of the EDFA were not studied such the nonlinearities due to high EDFA gain.

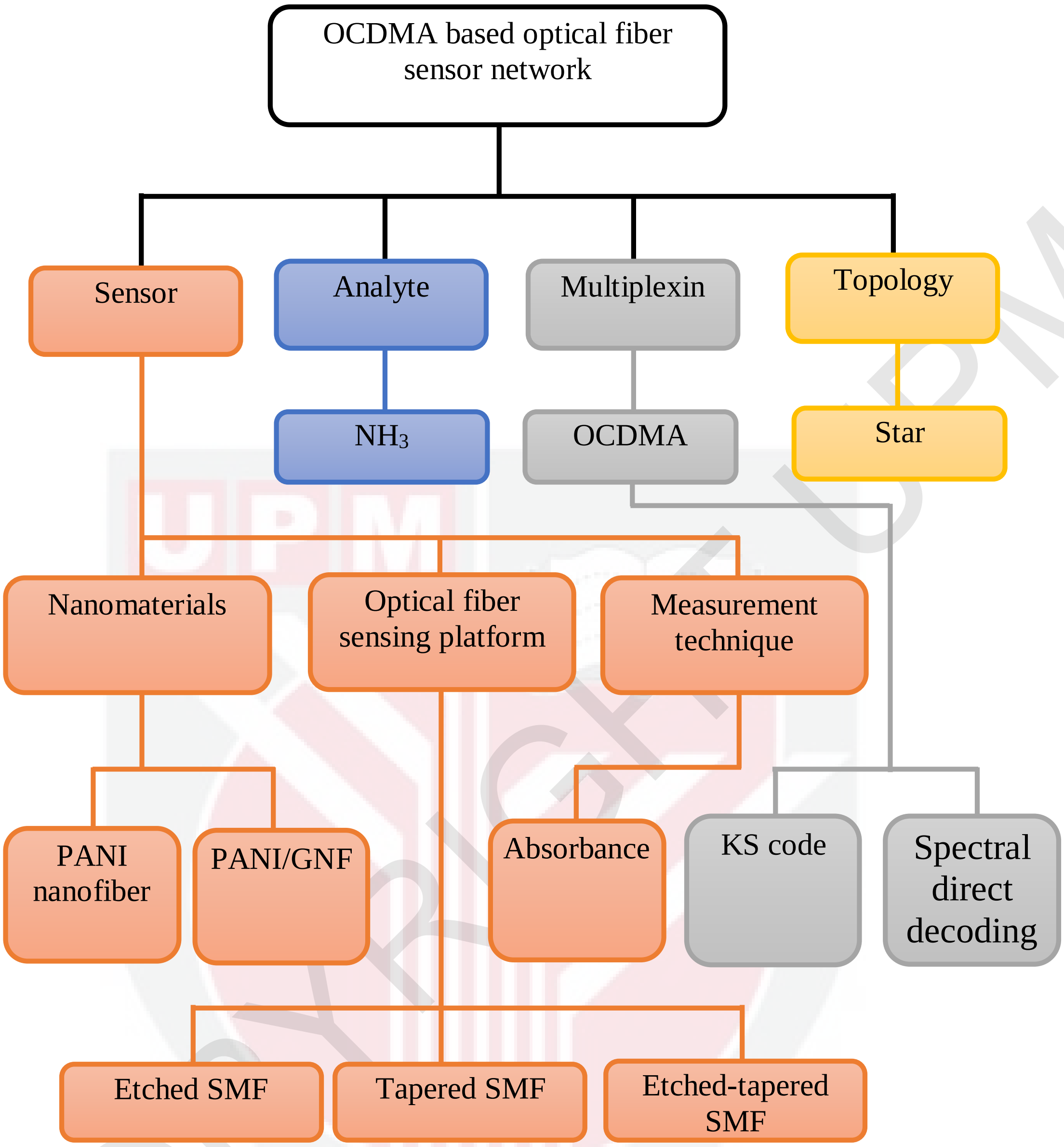

Figure 1.1 : Scope of PhD research work

1.5 Thesis Organization

This PhD research project reported in the thesis is consists of seven chapters. Chapter One provides a brief overview of the nanomaterial based optical fiber sensors for remote monitoring using optical sensor network with the problem statements and objectives. The rationales, theoretical background and review on the previous reported research findings related to this work are presented in Chapter Two. In Chapter Three, the modifications on the optical transducing platforms and their characterizations are highlighted. This chapter also elaborated in details the characterization of the nanomaterials employed as a sensing layer. The micro-characterization results were achieved through series of characterization techniques. Chapter Four describes the fabrication and performance investigations of different modified SMF sensors coated with PANI nanofiber. The optical sensing performance investigations were carried out in both visible and C-band wavelength ranges. Chapter Five highlights the fabrication processes for the etched-tapered SMF and the deposition of PANI/GNF nanostructured thin films onto the sensors. Chapter Six outlines the details of SAC\OCDMA based star optical fiber sensor network system consolidating SMF multiple point sensors. Remote monitoring results based on the demonstrated optical fiber sensor network system are discussed throughout Chapter Five and Six. This includes the use of the EDFA for signal enhancement. Finally, the research works are concluded in Chapter Seven. It presents a conclusion to the thesis by evaluating the key results with the project objectives and highlighting the author’s novel contributions while providing a glimpse into the future work for optical gas sensor network.

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