An active core fiber optic gas sensor using a photonic crystal hollow core fiber as a transducer

Venkata Satya Sai Sarma Tipparaju

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AN ACTIVE CORE FIBER OPTIC GAS SENSOR USING A PHOTONIC CRYSTAL
HOLLOW CORE FIBER AS A TRANSDUCER

By
Venkata Satya Sai Sarma Tipparaju

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AN ACTIVE CORE FIBER OPTIC GAS SENSOR USING A PHOTONIC CRYSTAL

HOLLOW CORE FIBER AS A TRANSDUCER

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An active core fiber optic gas sensing technique has been developed by using a photonic crystal (PC) hollow core fiber (HCF) as a transducer and a tunable diode laser as a light source for multi-gas sensing. The intrinsic optical absorption signal of an analyte molecule in the near infrared region is monitored for sensing C$_2$H$_2$, CO$_2$ and NH$_3$. Although the overtone absorptions are known to have low absorption cross-sections, this sensor can detect these gas components down to the parts-per-million (ppm) level by using a 1-meter hollow core fiber as a transducer. The sensitivity of this gas sensing technique can be improved by introducing periodic openings along the fiber, decreasing the hole diameter down to 0.5 µm and using a longer hollow optical fibers. Other advantages of this gas sensing technique include less interference, fast response and potential applications like high temperature, remote and corrosive gas sensing.

Key words: absorption spectroscopy, laser diode, hollow core photonic crystal fiber.
DEDICATION

I would like to dedicate this thesis to my parents, my brother Anil Kumar and to all family members and friends.
ACKNOWLEDGEMENTS

First of all, I would like to express sincere thanks to my research advisor and also my thesis committee chairman, Dr. Shiquan Tao for all his support, guidance and directions during my entire thesis work and also I would like to thank him for hiring me as a Research Assistant and for all the training he provided. I also thank Dr. David L Monts, graduate coordinator and also my committee member, for all his help, guidance, and valuable suggestions. Expressed appreciation is also due to other committee members Dr. Chuji Wang, and Dr. Chun Fu Su for their valuable time and suggestions. Finally, I would like to acknowledge my parents, my brother Anil Kumar and all my family members without their support this wouldn’t be possible.
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CHAPTER I
INTRODUCTION

The objective of this work was to design and characterize a laser diode-based gas sensor using a special kind of fiber called a photonic crystal hollow core fiber (PC-HCF). With the help of a distributed feedback (DFB) laser diode at the selected wavelength of 1531.2 nm the sensitivity and selectivity increases for a particular gas species with no observable cross response from other species. By tuning the temperature of the DFB laser diode, the gases, like C\textsubscript{2}H\textsubscript{2}, NH\textsubscript{3}, and CO\textsubscript{2}, can be detected with high sensitivity and fast response in the near-infrared region. Detection of these gases is indeed important for health, safety and environmental reasons. Extremely low levels of NH\textsubscript{3} in clean rooms may drastically deteriorate the performances of lithography process [1], sensors are also needed for environment monitoring to check NH\textsubscript{3} emissions from animal production facilities and automobile traffic [2] or in medicine to analyze breath NH\textsubscript{3} levels as a diagnostic tool [3], and for monitoring the NH\textsubscript{3} and CO\textsubscript{2} content in bioreactor’s vent gases [4]. These gases are not only pollutant gases, but also play an important role in many chemical processes. Detection limits based on this technique can detect their concentrations accurately with fast response. It can be used for industrial applications. In chapters I to V deals with instrumentation, physical and chemistry properties and near infrared, chapters II deals with the experimental details, results and discussions. The gas
detection using this method can sense down to the parts-per-million level with fast response time with optimized parameters such as hole diameter, periodic openings and length of the fiber, the sensitivity and response time can be improved significantly. This HC-PCF is very vulnerable to water and dust so it needs a gas membrane film, which allows only gas in and out, but prevents dust and water entering the HC-PCF. This sensor especially can be used for online monitoring at room temperature and at atmospheric pressures with high sensitivity and fast response.

*Photonic crystal hollow core fiber*

A photonic crystal fiber (PCF) contains microstructures like photonic crystals that change the propagation of light waves. In a PCF light is guided in a hollow core by these periodic array of microscopic air holes like circular array of holes in triangular shape in HC-PCF which are designed by special fabrication techniques, make the light to propagate at the center of the hollow core by multiple Bragg reflections or closely related to multiplayer mirrors but it is different from conventional optical fiber in which light is guided by total internal reflection. A prominent example of a photonic crystal is the naturally occurring are Butterfly wings and gemstone Opal [5]. PCF can be divided into two types based on their light guiding mechanism.

*Index-guiding fibers*

Index-guiding fibers are different from Bandgap fibers in which it contains a solid core at center surrounded by random arrangement of holes and light is guided by total
internal reflection. The total effective index of the cladding is less than reflection as in conventional optical fibers.

**Bandgap-guiding fibers**

Bandgap-guiding fibers different from Index-guiding in which it contains a hollow core at centre surrounded by periodic microstructures which results in a photonic bandgap that confines light inside a hollow core or in a core made of material with a refractive index lower than that of the effective index of the cladding. Here the total effective refractive index of cladding is greater than refractive index of hollow core so the light is not guided by total internal reflection. The light is guided by multiple Bragg reflections which results due to banggap effect. The wavelength depends on the bandgap. The guided light is only finite range of frequencies. The range is ±10 % of central wavelength of HC-PCF.

**Fabrication techniques**

**Stack-and-draw procedure**: - In this method silica capillaries of ~1mm diameter are stacked together and drawn at conventional drawing tower. In order to maintain the integrity of the circular holes the temperature is maintained at (~1900°C) as shown in the Figure 1 but less than conventional fibers in which temperature is maintained at (~2000°C). A polymer jacket is provided on outer surface to give mechanical strength and flexibility. These fibers are available in two different core sizes; one is by removing 7 cells and the other by removing 19 cells from the center [6]. The 19 cells give 20μm±2
core size and 7 cells gives 10.6µm fiber. The larger core gives lower loss, lower dispersion while the smaller core fibers provide a wider continuous operating wavelength band and support a smaller number of modes. In this experiment, a 19-cell fiber was used, which was purchased from Thorlabs Company.

Figure 1  Stack-and-drag fabrication technique

(a) Stack of glass tubes (b) runs at 1900°C (c) hollow core photonic crystal fiber; [http://www.sciencemag.org/cgi/content/full/299/5605/358](http://www.sciencemag.org/cgi/content/full/299/5605/358)

**Optical Properties**

**Transmission loss:** - The light loss of hollow core photonic crystal fibers is about 0.02 dB/km. The relative low loss of photonic crystal fibers can be used for achieving ultra sensitivity gas detection by using longer path lengths. The light loss in these fibers can be
reduced by enhancing the structural uniformity of the fiber in both transversal and longitudinal direction as well as by reducing material contamination and the surface roughness of the holes. For a 20 μm core size of hollow core photonic crystal fibers transmission loss is around 1.2dB/km at 1620 nm and loss of 13dB/km for a 10.6 μm at 1500 nm. In hollow core photonic crystal fibers, losses are mainly caused by leakage loss, coupling to surface and cladding modes, light scattering and while the limiting loss mechanism is due to surface capillary waves [7-8].

**Dispersion**: - Waveguide dispersion depends mainly on the core diameter and on the refractive index between the core and the cladding. Conventional optical fibers have small waveguide dispersion due to a low index between the core and the cladding. The dispersion characteristics of hollow core photonic crystal fibers can be altered by changing the pitch (\(\wedge\)) and air-hole size (d). This gives tailored waveguide dispersion. Because of these PCFs gives new properties. The effect of waveguide dispersion is particularly strong for PCFs with a high air-filling fraction (d/\(\wedge\)) about small dimensions. By properly choosing the parameters wavelength can be shifted from the near infrared to visible wavelengths [9].

**Birefringence and polarization-mode dispersion**: - Birefringence in optical fibers is defined as small variations in the symmetry of the fiber. It also defined as asymmetrical stress distribution along the fiber. It changes randomly along the fiber. Generally this phenomenon is called polarization-mode dispersion. It limits the data transmission in long distance communication systems. The PCF has large refractive index which enables
high whereas the fabrication technique gives a precise control over the cross-sectional index profile. The birefringence in PCFs is usually based on the cladding or periodic microstructures or the asymmetrical shape of the core. The birefringence in the PCFs is robust against temperature variations because of the single fabrication material. This unique property can be used in gyroscopes, interferometers and polarimetric sensors [7-9].

Non-linearity: - The Stack and draw fabrication procedure of hollow core photonic crystal fibers allows with a very small core diameter (10-20 µm) and hole diameter of (1-3 µm) with a high air-filling fraction. Due to this, the propagating modes can have very small effective mode areas compared with conventional fibers. Theoretically non-linear coefficient is inversely proportional to mode area. Since PCFs has small mode area, it has high non-linearites and also vice versa. This gives PCFs new possibilities in nonlinear optics with special dispersion properties. Furthermore, the mode area of PCFs can be wavelength dependent. This can be exploited in the realization of wavelength dependent non-linear effects [7-10].
CHAPTER II

GAS SPECTROSCOPY USING PHONIC CRYSTAL FIBER

One of the applications of photonic crystal hollow core fiber technology is gas sensing with high sensitivity and fast response. The PC-HCF fiber offers strong light confinement in the core and \( \sim 99.8\% \) of the light can propagate in a gas filled hollow core [11]. When a light beam interacts with the gas sample in the PCF fiber, the photons can excite molecules to high energy levels. Each level is quantized and consists of a combination of electronic, vibrational, rotational and spin energy levels. Consequently, molecules can only absorb discrete amounts of energy, resulting in the loss of light at specific absorption wavelengths. Each molecular species has a unique absorption spectrum from which they can be identified. A typical absorption spectrum consists of absorption bands that again comprise a number of closely spaced absorption lines. Generally, in absorption spectroscopy, light transmission through a sample is measured as a function of frequency or wavelength. This can be done by utilizing a monochromatic tunable laser source and a near infrared photo detector. The molecular concentration is subsequently retrieved from the measured absorption. This is given by Beer-Lambert law.
Beer-Lambert Law

The relationship between the percent transmission of the incident radiation reaching the detector and the concentration of the component of interest is called as the Beer-Lambert law or Beer-Bouguer law or simply Beer’s law and it is given by

$$\log_{10}(I/I_o) = -abc \quad \text{or} \quad I = I_o e^{-abc} \quad (1)$$

where $a$ is the absorption coefficient, $b$ is the path length and $c$ is the concentration of the sample or more specifically, the absorption coefficient at frequency $\nu_0$ is given by

$$a (\nu - \nu_0) = \frac{P}{k_B T} * S * g(\nu - \nu_0) \quad (2)$$

where $I$ represents the amount of radiation transmitted by the solution and $I_o$ represents the total energy of the incident beam, $a$ is absorption coefficient whose value depends on the absorbing molecule at a particular frequency $\nu$, $b$ is the path length, $c$ is the concentration of the absorbing molecule, $p$ is pressure, $T$ is absolute Temperature, $k_B$ is Boltzmann’s constant ($1.38065 \times 10^{-23} \text{ m}^2 \text{ kg s}^{-2} \text{ K}^{-1}$), $S$ is the line strength of the given line, $g(\nu - \nu_0)$ is the area-normalized lineshape function at frequency $\nu_0$. The term $(I/I_o)$ represents the percent transmission of the absorbing material and we can express the Beer-Lambert relationship as

$$\log t = -abc \quad \text{or} \quad \log (1/t) = abc \quad (3)$$

where ‘$t$’ is the transmittance, which is given by $t = (I/I_o)$ \quad (4)

It should be noted that the concentration of a substance is not related in linear manner to the percent transmission. For analytical calculations, a simplified equation is obtained by

$$A = -\log (I/I_o) = -\log t = \log (1/t) \quad (5)$$

Therefore the Beer-Lambert expression becomes
The absorbance \( A \) is a quantity measurable directly from a spectrophotometer simply by using an absorbance scale rather than a transmission scale [12]. Absorption spectroscopy can be used for selective gas sensing. The gases in near-infrared regions have low absorption cross-sections. Due to weaker strengths results in a reduced sensitivity. In order to achieve higher sensitivity multi-pass technique is required to increase signal strength. The availability of telecommunication tunable laser diodes in 1.5 \( \mu \)m region has led to increasing interest in the use of overtone bands. By using long optical path length with photonic crystal hollow core fiber the high sensitivity and fast response can be achieved.
CHAPTER III

PHYSICAL AND CHEMICAL PROPERTIES

Ammonia gas

Under standard conditions ammonia is a colorless gas, with a pungent odor. Ammonia consists of one nitrogen atom and three hydrogen atoms and chemical notation is NH₃ and its molecular weight (M= 17 gram/mole) making it a good deal lighter than air, (M = 29 gram/mole). So, it tends to rise when released. Density of ammonia is 0.77 gram/liter at 0ºC. Its critical temperature is 132.4ºC. It is very soluble in water. Under standard conditions, the saturated solution is about 30% ammonia and is a powerful cleaning agent. Household ammonia contains 5 to 10% of ammonia diluted in water. Under 1 atm, pure ammonia melts at -77.7ºC, boils at –33.4ºC. High amounts of ammonia are used in manufacture of explosives, fertilizers and nitric acid. The salts of ammonia are used for dry cells, washing powders, in the manufacture of rubber goods, and photographic films. It acts as very good refrigerant. Ammonia is very toxic to aquatic animals, even at very low concentrations. Ammonia in small amounts in air is extremely irritating to eyes, throat, and breathing passages. It causes mild irritation sometimes destruction of eyes. Long exposures ruptures respiratory tissue and Vapors can cause coughing, and breathing difficulties. It also affects the skin from mild irritation to severe burns, damage depends on length of exposure and concentration [13-14].
Carbon dioxide gas

Carbon dioxide is a colorless, odorless gas. It consists of one carbon and two oxygen atoms. Its chemical formula is CO₂. The molecular weight is 44.01 gram/mole. Its melting point is -55.6°C, Boiling point is -78.5°C and density is 1.977 kg/m³. Carbon dioxide is neither combustible nor a supporter of combustion. At 1 atmosphere and temperature of 15°C, 1 volume of water dissolves about 1 volume of gas. Air containing more than 10% CO₂, if breathed, is life threatening. It is 1.5 times heavier than air. Carbon dioxide is a slightly toxic, odorless and colorless gas. It is stable and inert. It is very good refrigerant. Carbon dioxide is commercially used in manufacture of beverages, fire extinguishers, and in making dry ice. It forms dry ice at -78.5°C. Dry ice is especially used in shipping ice creams and perishable foods and it has advantage over ice that there is no water to dispose of, and it is much lighter than ice. The primary health dangers of carbon dioxide are asphyxiation, frostbite, kidney damage, or coma. This is caused by a disturbance of the chemical equilibrium of the carbonate buffer. It is dangerous when carbon dioxide concentrations increase or decrease [15].

Acetylene gas

Acetylene is a colorless and almost odorless gas when pure. It consists of two carbon atoms and two hydrogen atoms. The molecular formula is C₂H₂. Its molecular weight is 26.03 gram/mole. Acetylene gas with impurities has a strong garlic odor. It is unstable, highly combustible, and produces a very hot flame when combined with oxygen. The principal raw materials to prepare acetylene gas are coal and limestone. It
can also be prepared by partial combustion of methane with oxygen. It is highly unstable and explodes while compressed. Its melting point is \(-84^\circ\text{C}\), boiling point is \(-80.8^\circ\text{C}\) and density is 1.096 kg/m\(^3\). It is chiefly used for cutting and welding. It is also used for general anesthesia under the trade name narcylene. If inhale, it caused dizziness, headache and nausea. Impurities in acetylene gas are toxic and sometimes even fatal [16].

**Ammonia structure**

Ammonia has symmetric top structure with one central N-atom above a tripod of three H-atoms. The A-axis corresponds to the figure axis of a symmetric top, and thus is also the main axis of symmetry. Symmetric tops have two equivalent moments of inertia, both of which are different from the third moment:

\[
I_A \neq I_B = I_C, \quad I_A \neq 0
\]  

(7)

The rotational constants in cm\(^{-1}\) are related to the moment of inertia by

\[
A_0 = \frac{\hbar}{8\pi^2 I_A c}, \quad B_0 = \frac{\hbar}{8\pi^2 I_B c}, \quad C_0 = \frac{\hbar}{8\pi^2 I_C c}
\]

(8)

Typically, the equivalent moments of inertia for symmetric top molecules are described simply as \(I_B\), and the main axis moment of inertia is \(I_A\). The relative magnitudes of the inertial moments or rotational constants can be used to further distinguish the tops as prolate or oblate:

Prolate: \(I_A < I_B = I_C; \quad A > B = C\)

(9)

Oblate: \(I_A > I_B = I_C; \quad A < B = C\)

(10)

Thus, ammonia is an oblate symmetric top. Since ammonia has two main directions of rotation, it has two quantum numbers to describe rotational energy, \(J\) represents the total
angular momentum (0,1,2…) and K represents the angular momentum about the A-axis (in other words, K represents the orientation and direction of rotation) [17].

**Carbon dioxide structure**

CO₂ is a linear triatomic molecule. The moment of inertia about the A-axis is approximately zero, Iₐ = 0, and the other moments are equal, I₃ = I₅. The moment of inertia can be related to a molecule’s rotational constant via the following relation

\[ B₀ = \frac{h}{8\pi²I₃c} \]  

(11)

Since CO₂ is linear and symmetric, it does not have a permanent dipole moment. Thus, CO₂ is spectroscopically active in the infrared when a dipole is induced due to bending or asymmetric stretching. CO₂ has four classically described vibrational modes of which two bending modes are degenerate. The two stretching modes, asymmetric and antisymmetric, are parallel vibrations, since the vibrations are parallel to the main symmetry axis, and the bending modes are perpendicular, since they induce changes in the molecule that are perpendicular to the main symmetry axis. Only the vibrations that induce a dipole moment are spectroscopically active in the infrared [17].

**Acetylene structure**

Acetylene is linear symmetric molecule. Each carbon is Sp-hybridized. The two sp carbon atoms in acetylene are joined by triple bond with an angle of 180°. Two pi bonds are perpendicular to each other. There is a large amount of charge density in the region of C-C bond. Since this comes under linear polyatomic molecule, the moment of inertia is similar to the moment of inertia about the A-axis is approximately zero, Iₐ = 0,
and the other moments are equal, $I_B = I_C$. The molecular constant $B$ is given by equation (11). Acetylene has 7 normal modes, in which four are Raman active and three are Raman inactive [17].
CHAPTER IV
INFRARED SPECTROSCOPY

Infrared (IR) spectroscopy is the spectroscopy which deals with the infrared of the electromagnetic spectrum. The infrared region is subdivided into three regions: Near infrared region (13,300-4000 cm\(^{-1}\)), mid infrared region (4000-400 cm\(^{-1}\)) and far-infrared (400-20 cm\(^{-1}\)). For different chemical compounds, the rotational and vibrational levels are different, giving rise to different transitional fingerprints in the infrared. The far infrared region has low energy and is useful for detecting those molecules which exhibit rotational transitions. The mid-infrared is useful for detecting, rotational-vibrational transitions. An infrared beam is passed through a sample and the energy absorbed at each wavelength is recorded. Common incandescent or quartz halogen light bulbs are most often used as broadband sources of near infrared radiation (NIR). In fact light bulbs are the most common radiation sources for the NIR-based analytical applications. It is becoming more common to employ laser diodes as well [18]. In this work the 1531.2nm distributed feedback (DFB) was utilized.

Ammonia

Strong absorption lines of ammonia occur around 1.5 µm. The Table 1 show below includes six features of Ammonia at 1531 nm [17]. Among these transitions, the one at 1531.7 nm is the most suitable overall, because it has significant with other
transitions. The one at 1527 nm offers the best choice due to having the least interference from combustion species. So these two lines were selected for this research. The commercially available laser diodes operate at 1527.6 nm and 1531.2 nm. These two laser diodes are selected for my research in designing near infrared NH₃ sensors. Most of these transitions are due to \( \nu_1 + \nu_3 \) and \( 2\nu_3 \) combination and overtone bands, though other bands such as the \( 2\nu_1, \nu_1 + 2\nu_4 \) are also present [19-20].

Table 1  Six absorption lines of ammonia around 1530 nm

<table>
<thead>
<tr>
<th>Line. No.</th>
<th>Wavelength(nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1531.7</td>
</tr>
<tr>
<td>2</td>
<td>1527.0</td>
</tr>
<tr>
<td>3</td>
<td>1522.4</td>
</tr>
<tr>
<td>4</td>
<td>1516.0</td>
</tr>
<tr>
<td>5</td>
<td>1515.2</td>
</tr>
<tr>
<td>6</td>
<td>1497.4</td>
</tr>
</tbody>
</table>

**Carbon dioxide**

CO₂ is linear triatomic molecule. The number of fundamental vibrational modes is four, of which the two bending modes are degenerate. The two stretching modes, symmetric and antisymmetric are parallel, since the vibrations are parallel to the main symmetry axis. The bending modes are perpendicular, since they induce changes in the
molecule that are perpendicular to the main symmetry axis. Only the vibrations that induce a dipole moment are spectroscopically active in the infrared. The vibrational overtone and combination band responsible for transitions near 1.5 μm for CO₂ is 2ν₁ + 2ν₂ + ν₃ [21].

**Acetylene**

Acetylene has a large number of rotational and vibrational transitions between 1510 nm and 1540 nm as shown in Figure 2 [22]. According to classical theory, if the frequency of infrared light matches with molecular vibrational frequency or resonant frequency or normal modes, maximum absorption of light occurs. The absorption spectrum with respect to frequency or wavelength is called infrared spectrum. Thus an absorption spectrum consists of molecular vibrational frequencies.

![Figure 2](image.png)

Figure 2  Spectrum of v₁ + v₃ combination band of acetylene

Figure taken from [22]
The $\Delta J = -1$ and $\Delta J = +1$ transitions are referred to as P and R branch lines respectively, as shown in Figure 3. Simultaneous existence of rotational and vibrational motions is due to molecular vibrations change the moment of inertia of the molecule. In polyatomic molecules, one photon may also excite more than one vibrational mode simultaneously. Bands arising from these transitions are called “combination” bands.

![Diagram showing P and R branch lines in transition lines of acetylene.](image)

**Figure 3** P ($\Delta J = -1$) and R ($\Delta J = +1$) branches in transition lines of acetylene.

Figure taken from Ref [22]

Generally the number of normal modes is given by $3N-6$, where $N$ is number of atoms of a molecule. For a linear molecule, the number of fundamental modes is given by $3N-5$. Since acetylene molecule is linear, acetylene has 7 normal modes of vibration, in which five have independent frequencies as shown in Figure 4. The absorption lines
around 1.5 μm are due to the simultaneous excitation of \( \nu_1 \) and \( \nu_3 \) that arise primarily due to CH stretching motions.

<table>
<thead>
<tr>
<th>Mode</th>
<th>Description</th>
<th>Normal Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \nu_1 )</td>
<td>Symmetric C-H stretch</td>
<td>[\text{H-C\equiv C-H}]</td>
</tr>
<tr>
<td>( \nu_2 )</td>
<td>Symmetric CC stretch</td>
<td>[\text{H-C\equiv C-H}]</td>
</tr>
<tr>
<td>( \nu_3 )</td>
<td>Asymmetric C-H stretch</td>
<td>[\text{H-C\equiv C-H}]</td>
</tr>
<tr>
<td>( \nu_4 )</td>
<td>Symmetric bend</td>
<td>[\text{H-C\equiv C-H}]</td>
</tr>
<tr>
<td>( \nu_5 )</td>
<td>Asymmetric bend</td>
<td>[\text{H-C\equiv C-H}]</td>
</tr>
</tbody>
</table>

Figure 4  Fundamental vibrational mode of acetylene.

Figure taken from Ref [22]
Laser is an abbreviation for Light amplification by stimulated emission of radiation. It is an optical source that emits photons in a coherent beam. Lasers typically emit near-monochromatic emits a single color or single wavelength. The term “lase” means “to produce laser light” or possibly “to apply laser light to.” In analogy with optical lasers, a device that produces any electromagnetic radiation in a coherent state is called a “laser”. In most cases, “laser” refers to a source of coherent photons. This contrasts with common light sources, such as the incandescent light bulb, which emit incoherent photons almost in all directions usually over a wide spectrum of wavelengths.

Lasers are available from visible, ultraviolet to infrared regions. The different types of lasers are

- Gas lasers
- Chemical lasers
- Dye lasers
- Metal-vapor lasers
- Solid-state lasers
- Semiconductor lasers

**Distributed feedback laser diode (DFB LD)**

The DFB LD comes under the category of quantum cascade laser of semiconductor diode lasers. It consists of several alternating two different kinds of
semiconductors, such as GaAs/AlGaAs or AlInAs/InGaAs, forming a quantum heterostructure. The layers are grown on a substrate, using a technique called molecular beam epitaxy. The 14-pin of a DFB LD is shown in Figure 5. The emitted wavelength depends on the thickness of the layers or sometimes on the layer materials itself. This is a great advantage over diode lasers, whose wavelengths depend primarily on the band gap of the given material. The wavelengths that are available to quantum cascade lasers are in the range of 3.5 – 16 µm covering the mid-infrared range. The reflection produced by a periodic structure or a structure called distributed feedback, abbreviated to DFB along the active region is built on top of the laser crystal to prevent it from emitting at other than the desired wavelength. On the other hand, a similar structure which is separated from the active region is called a distributed Bragg reflector. The whole cavity of a DFB laser consists of a periodic structure that produces a phase shift in middle, which acts as a distributed reflector in the wavelength range of laser action and that contains a gain medium. This can be achieved by a periodic corrugation of one of the two surfaces of the action regions which results in effective index variation. Due to this variation, distributed reflectivity arises due to Bragg scattering of the laser beam which in turn acts as a distributed phase grating. The distributed reflectivity DFB lasers are Fabry-Perot single mode lasers, containing an integrated grating structure that results in single mode emission with a precise wavelength and with extremely narrow line width. Most distributed-feedback lasers are either fiber lasers or semiconductor lasers. DFB laser use diffraction gratings rather than mirrors to select the resonance and oscillation in the cavity. In order to obtain maximum feedback the ratio between selected wavelength to
the effective refractive index must be equal to spatial period. Even though it has multiple axial modes but only one mode is dominated. Thus, emitted spectrum of a DFB laser diode generally consists of single longitudinal mode. This structure is thus basically the direct concatenation of two Bragg gratings with internal optical gain. It produces a finer range of wavelengths [23-27].

![DFB Laser Diode](image)

Figure 5  Distributed Feedback laser diode

*Butterfly laser diode mount*

The DFB laser diode is fixed on the Butterfly laser mount as shown in Figure 6. The Thorlabs LM14S2 is a universal laser mount specifically designed for Butterfly laser diodes that have integrated thermal electric coolers (ITC) and thermistor sensors. When it is used with a ITC 502 laser diode combo controller, the laser diode can be controlled using precise temperature control for wavelength stability and temperature tuning.
The ITC 502 laser diode combo controller as shown in Figure 7 is used to drive the DFB laser diode by providing the desired temperature and current by using a 14-pin butterfly laser diode mount. The ITC 502 provides current and temperature control in one unit. It provides a maximum laser drive current range of ±200 mA or ±1A, and a TEC drive current of up to ±2A or ±4A. The features are low current noise and low temperature drift. The laser diode protection includes soft start, over temperature protection, laser current limit, interlock, TEC current limit, temperature window protection, open circuit detection, and “no sensor” detection, short circuit when laser is in off condition. This instrument can also be operated remotely to enhance speed and control capabilities through interface program either in C++ or in Lab View. The ITC is connected to USB computer port by using GPS-USB-HS cable. The front panel is divided into two major sections. The temperature control section on the left panel shows set
temperature, the actual temperature, the TEC current, the TEC voltage, the TEC current limit, or the set temperature. The current source section on the right shows Laser current, laser voltage, monitor current, optical power, current limit.

Figure 7   ITC 502 IEEE laser diode combo controller

_Three-axis nano piezo electric controller_

To align the hollow core photonic crystal fiber to laser beam, a three-Axis Nano Piezo Electric Controller was used as shown in Figure 8. By adjusting the differential micrometers in X, Y and Z directions, adjustments can be made. Apart from this, it also contains piezo electric controller with SMC connectors to provide nanometric positioning down to sub-micron resolution.
Photodiode amplifiers

A Photodiode is a p-n junction semiconductor diode operated with an applied reverse-bias voltage in which it offers greater resistance. The resistance is reduced when light of an appropriate frequency falls on the junction or the mobile electrons flow across the depletion layer between the valency band and conduction band and electrons flow across the junction generates a photocurrent or voltage. The materials used to make a photodiode are critical to defining its properties, because only photons with sufficient energy to excite an electron across the material’s bandgap will produce significant photocurrents. These photodiodes can make to detect different electromagnetic regions by choosing different kind of materials. Table.2 shows which materials can be chosen for selected wavelength ranges.
Table 2  Kinds of material used for photodiodes to detect different wavelength ranges

<table>
<thead>
<tr>
<th>Material</th>
<th>Wavelength range (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>190-1100</td>
</tr>
<tr>
<td>Germanium</td>
<td>800-1700</td>
</tr>
<tr>
<td>Indium Gallium Arsenide</td>
<td>800 - 2600</td>
</tr>
<tr>
<td>Lead Sulfide</td>
<td>&lt;1000-3500</td>
</tr>
</tbody>
</table>

**Features**

**Responsivity:** - It is defined as the ratio of generated photocurrent to incident light power or the ratio of the number of photons generated carriers to incident photons.

**Dark current:** - It is defined as current through the photodiode in the absence of any input optical signal is called Dark current. It includes photocurrent generated by background radiation and the saturation current of the semiconductor junction.

**Noise-equivalent power (NEP):** - NEP minimum input optical power to generate photocurrent equal to the root mean square noise current in 1 Hz bandwidth. The NEP is roughly the minimum detectable input power of a photodiode. The detectivity is inversely proportional to NEP and the specific detectivity ($D^*$) = $D\sqrt{A}$, where A is the area of the photodetector.
Comparison with photomultipliers

The advantages over photomultipliers are low noise, having long lifetime, compact and light weight, no high voltage required, quantum efficiency is typically 80%, and spectral response is from 190 nm to 1100 nm and. The disadvantages are having small area, response time is very slow, less sensitivity, and no internal gain except avalanche photodiodes [28-32].

Photodiode amplifier 400

In this work, an amplified photo detector for infrared region was used. It is an amplified, switchable-gain. The PDA400 is having active diameter of 1mm and the response is from 800 –1750 nm. The output voltage is 0 to 10V. A five-position rotary switch allows changing the gain in 10dB steps. The light to voltage conversion can be estimated by [28-35].

\[
\text{Output (n volts/watt)} = \text{transimpedance gain}\ (\text{V/A}) \times \text{responsivity}\ (\text{A/W})
\] (12)
CHAPTER VI
EXPERIMENTAL MEASUREMENTS AND ANALYSIS

With the help of an air-guiding photonic crystal hollow Core (~18 μm diameter) fiber and semiconductor distributed feedback laser diode (DFB) in near infrared region at selected wavelengths 1531.2nm and 1527.6nm as a laser source, a simple, robust, portable high sensitivity gas sensor at atmospheric pressures was designed. By tuning the temperature of the DFB laser diode, which in turn changes the wavelength and by carefully analyzing the absorption band lines of the gases, C₂H₂, NH₃, and CO₂ gases were detected with fast response, high sensitivity and selectivity for a particular gas species. Absorption spectroscopy can be conveniently applied for selective gas sensing, diagnostics, process and emission control and trace gas monitoring. In recent years, a need of high sensitivity, simple and continuous determination portable gas sensors with fast response and reliability has been identified. This can be achieved with the availability of tunable laser diodes and low loss optical waveguides like hollow core photonic crystal fiber (HC-PCF) in the 1550 nm region have led to an increasing interest in the use of overtone bands which exhibit weaker transitions. It is also possible to detect different gases without any interference with a single wavelength laser source. The detection of gases like NH₃, CO₂, C₂H₂ is indeed important for health, safety and environmental reasons. Absorption spectroscopy techniques detect their concentrations accurately with
fast response at atmospheric pressure. The above gases show rotational-vibrational overtone and combination bands in near-infrared region. In this work, a pigtailed DFB 1531.2nm laser diode and by tuning the laser diode, detected NH₃, CO₂, and C₂H₂ diluted in N₂. The minimum detectable absorption with 3σ convention is of 171 parts-per-million per 1 meter (ppm-m), 32-ppm–m, and 18 ppm–m and with pigtailed DFB 1527.6nm laser diode, detected NH₃ gas and the minimum detectable absorption of 482 ppm-m.

**Experimental set-up**

In order to achieve high sensitivity with fast response a hollow core photonic crystal fiber (PCF), with core diameter of 18.44μm (HC19-1550-01 from Thorlabs) of path length 1m was used. More than 98% of the optical power propagates in the hollow core or in the holes of the cladding that run along the entire fiber length, which in turn can prevent the escape of light from a hollow core. This is different from conventional optical fibers in which light is guided by total internal reflection. In addition, the PC-HOF is made from quartz; a material can tolerate high temperatures and is corrosive resistance. These features make PC-HOF especially useful for applications for sensing corrosive gases at high temperatures. The holes can be filled by a gas, which can alter the attenuation and non-linear properties of the fiber. In this fiber, the diameter of hole region is 64.67 μm and diameter of each hole is 3.67 μm as shown in Figure 9. The laser sources selected for this experiment are semiconductor InGaAsP pigtailed DFB 1531.2nm and 1527.6nm laser diodes with a power of about 20mW from Thorlabs part numbers DFB1531.12-20 and DFB1527.22-20 respectively. The temperature tuning rate is about 0.15 nm/°C and 0.003 nm/mA where the wavelength increases with an increase in
temperature. In these experiments, the temperature of DFB laser diode varied from 15°C to 35°C, which is equivalent to 1529.7 nm to 1532.7 nm with central wavelength 1531.2 nm laser diode and 1526.1 nm to 1529.1 nm for central wavelength 1527.6 nm laser diode. The experimental setup is shown in the Figure 10. In order to tune the temperature and current of the DFB laser diode, a benchtop laser diode and temperature controller was used of Thorlabs part numbers ITC 502-IEEE. The laser diode was mounted on the Universal 14-pin butterfly laser diode mount with part number LM14S2 respectively. To control this unit and to change the temperature remotely a software program was written in C++. For better alignment of the laser beam with the HC-PCF, a nano-Max positioning unit provided with a 20 μm travel range with a positional resolution of 20 nm was used. To collimate the laser, a 20X microscope was used as shown in Figures 10 & 11. To control the motion of the nano-Max in XYZ directions 3-Axis piezo controller unit from Thorlabs (part number MDT630A) was used.

Figure 9  Scanning electron microscope of a HC-PCF
One end of the HC-PCF is fixed to this system so that the laser beam exactly impinges at the centre of PCF core. The distal end is properly aligned to a 600 μm multimode fiber for the wavelength range 400-2200 nm Thorlabs, part number BFL48-600 in a T-shaped electrode. The gap between the HC-PCF fiber and the 600 μm is approximately 50 μm. The other end of the multimode fiber is connected to the PDA 400 near-infrared photodiode to detect the light signals. The output terminals of the photodiode are connected to the data acquisition card (DAQ) from National Instruments which in turn is connected to a laptop computer. The signals from the photodiode are converted into voltage signals by the DAQ card. To read the signals, a Labview software program was

Figure 10  Experimental set-up arrangement
used. The gas is feed in at the T-shape end of the electrode. A gas sample from a high-pressure gas cylinder was supplied via a two-stage regulator to the perpendicular port of the “T” electrode holder for introduction of the sample gas into the PC-HOF. The exit pressure of the two-stage gas regulator was adjusted to be 20 psi. The gas continuously flows into the PC-HOF through the gap between the hollow fiber and the silica optical fiber. The gas flowed through the hollow optical fiber escapes into the air from the distal open-end of the PC-HOF. The resulting signal was compared to the detected light intensity with nitrogen flowing through the PC-HOF. Therefore, the output signal of the sensor is a ratio of the output voltage signals, obtained with nitrogen and with the gas of interest \((V_{N2}/V_{sample})\). Three gas samples (1 vol.% \(\text{C}_2\text{H}_2-N_2\), 1 vol.% \(\text{NH}_3-N_2\), 1 vol.% \(\text{CO}_2-N_2\)) have been used to evaluate the sensor response. A temperature scan was
performed for each gas sample to determine the optimum operation temperature, and hence the wavelength of the laser diode, at which the PC-HOF filled with the gas sample gives the lowest intensity signal which corresponds to maximum absorption. The photons can excite molecules to high energy levels. Each level is quantized and consists of a combination of vibrational, rotational and spin energy levels. Consequently, molecules can only absorb discrete amounts of energy, resulting in the loss of light at specific absorption wavelengths. Each molecular species has a unique absorption spectrum by which they can be identified. This sensor has been calibrated for sensing the three gas compounds. In such a calibration experiment, the ITC-502-IEEE laser diode controller was set to the optimum temperature (wavelength) for a gas compound to be detected. A gas sample with high analyte concentration (1%) was diluted with pure nitrogen using homemade gas dilution system made to prepare samples of lower analyte concentrations. In the gas dilution system, the flow rate of stock gas sample (1%) and pure nitrogen was controlled by two gas flow meters. The two gas lines merged into one gas flow and produced a mixed gas flow. The concentration of the analyte compound in the obtained gas flow is controlled through changing the flow rate of the two gas flow meters. Because the central hole of the PC-HOF is so small, and because the flow rate of the two gas lines is too high, the mixed gas flow was split into two gas flow tubes after mixing. One of the gas tubes is connected to the gas inlet port of the “T”-shaped electrode holder. The distal end of the second tube is immersed 30 cm into water, which allows gas bubbles to escape into the surrounding air. The second tube was immersed deep into the water, creating a positive gas pressure at the tube connected to the gas inlet port of the “T”-connector so
that gas flowed through the central hole of the PC-HOF. This gas dilution system can dilute a stock gas at a high flow rate using a regular gas flow meter and deliver the mixed sample gas through the PC-HOF at a very low flow rate.

The experiment for each gas consists of three steps:

1. Identify the spectrum of the gas and identify the strong absorption lines
2. Calibration of the gas at the chosen strong absorption line
3. Time scan of the gas

**Temperature scan of gas**

The temperature of the DFB laser diode was varied from 15°C to 35°C remotely using our software program. The Lab view program was used to record the spectrum and identify the strong absorptions at particular wavelengths for each gas. Procedure was repeated several times for each gas. The lines we observed are the same reported in published papers results [36-42]. In the results we obtained, there was a background signal variation in all the spectra. The reasons are, due to the variation in laser diode intensity during change of temperature from 15°C to 35°C, interference effects, power variations, variation of the common mode laser power, and collision of molecules with the holes of the HC-PCF. There was a slight mismatch occurred when combining the temperature data from our C++ program and the voltage data from the LabView program. The results are shown in Figures 12, 13, 14 & 15 respectively.
Figure 12  Temperature scan for ammonia gas with DFB laser diode near 1531.2 nm

Figure 13  Temperature scan for ammonia gas with DFB laser diode near 1527.6 nm
1.39 Acetylene spectrum with 1531.2 nm laser diode

Figure 14 Temperature scan for acetylene gas with DFB laser diode near 1531.2 nm

1.9 Carbon dioxide spectrum with 1531.2 nm laser diode

Figure 15 Temperature scan for CO₂ gas with DFB laser diode near 1531.2 nm
**Calibration of gases**

**Ammonia gas:** Strong absorption lines were identified with both the diode lasers. For the 1531.2 nm diode laser, the strong NH$_3$ absorption is at 1531.7 nm. For the 1527.6 nm diode laser, the strong ammonia absorption is at 1526.49 nm. These lines are in agreement with the earlier reported results. Calibration was performed at these wavelengths [36-43]. The results are shown in Figures 16, 17, 18 & 19 respectively.

![Ammonia calibration at 1531.7 nm using 1531.2 nm laser diode](image)

**Figure 16**  Calibration of ammonia gas at 1531.7 nm
Ammonia calibration at 1526.49 nm with 1527.6 nm laser diode

Figure 17  Calibration of ammonia gas at 1526.49 nm

Ammonia calibration curve - 1531.2 nm laser diode

Figure 18  Calibration curve of ammonia gas at 1531.7 nm
Figure 19  Calibration curve of ammonia gas at 1526.49 nm

**Carbon dioxide gas:** - An absorption line was identified at 1530.45 nm. The published wavelengths line is 1531.6nm [43-44]. The results are shown in the Figures 20 & 21.

Figure 20  Calibration of CO₂ at 1530.45 nm
**Acetylene gas**: Absorption lines were identified at 1531.6 nm with DFB laser diode 1531.2 nm. These absorption lines are the P(9) line at 1530.43 nm and the P(11) line at 1531.6 nm [45-51]. The calibration was carried out at 1531.6 nm as shown in Figure 22. The resulting calibration curve is shown in Figure 23.

**Figure 21** Calibration curve of CO₂ at 1530.45 nm

**Figure 22** Calibration of acetylene gas at 1531.6 nm
Acetylene gas calibratin curve at 1531.6nm

\[ y = 4E-05x - 0.0205 \]

\[ R^2 = 0.9936 \]

**Figure 23**  Calibration curve of acetylene gas at 1531.6 nm

**Response time of the sensor**

The response time of these sensors is defined, as the time required for the gas to diffuse into/out of the holes of the HC-PCF. Since the holes are very small and irregular in shape, the fact that the wall affects the molecules must be considered. Our experimental result shows the response time for acetylene gas to diffuse into the holes is 3.38 min and to diffuse out is 10.91 min as shown in Figure 24. For ammonia gas, the time required to diffuse into the holes is 37min. About 12.6 min is required for upto 90% of the ammonia gas to diffuse out completely, and for remaining it is around 105 min as shown in the Figure 25.
Figure 24  Acetylene gas response time before modification of experimental setup

Figure 25  Ammonia response time
The expected response time should be immediate, but because a 2.5 m-length of connecting tube was used from the gas cylinder to the HC-PCF fiber, the response time is higher. The response times for acetylene gas by using a very small length of connecting tube and were around 34 sec, 14 sec as shown in Figure 26.

![Acetylene gas response time](image)

Figure 26  Acetylene gas response time after modification of experimental setup

In the case of ammonia gas, the response time is higher than that of acetylene gas. This is because of the adsorption effects of the polar ammonia molecules onto the walls of the HC-PCF [52] as shown in the Figure 25. According to the theoretical [53] for a 1 m length, the time for C₂H₂ to reach 90% of initial concentration is 200 min if both ends kept open. They also stated that for a response time of ~1 min; the length of the fiber
should be less than 7cm. In order to achieve higher sensitivity with a reasonable response time, the periodic openings along the sensing fiber need to be introduced as shown in Figure 27. To achieve further sensitivity, the optimum hole diameter of the HC-PCF should be 0.5 μm but in my experiments the hole size was 3.67μm.

![Sensing PCF with periodic openings](image.png)

Figure 27  Sensing PCF with periodic openings

By increasing the path length, decreasing the hole diameter, and introducing the periodic openings along the sensing fiber and optimizing all these parameters, it is possible to achieve higher sensitivity with reasonable response time for this sensor.

**Limitations of this HC-PCF sensor**

Hollow core photonic crystal fibers are very vulnerable to dirt and water. It needs a special gas membrane that is permeable only to the gas, but not to dust or water. Currently the cost of HC-PCF is expensive.
CHAPTER VII

CONCLUSION

In conclusion, an optical fiber gas sensor for multi-gas sensing has been built using a 1-meter of HC-PCF as a transducer, a tunable diode laser as a light source, and a near-infrared photodiode as a detector. The intrinsic optical absorption signals of C\textsubscript{2}H\textsubscript{2}, NH\textsubscript{3} and CO\textsubscript{2} in the NIR region has been monitored for sensing these gases. The preliminary results using a 1-meter HC-PCF as a transducer indicate that this sensing technique can be used to continuously monitoring trace gas components down to parts-per-million levels [54]. The advantage of this technique is that it can detect low absorption cross-section gases with high sensitivity and fast response for continuous monitoring. By optimizing parameters, such as increasing the path length, decreasing the hole diameter, and introducing periodic openings along the sensing fiber, it will be possible to achieve ultrasensitive with a fast response time. Other advantages of this sensing technique include less interference, fast response and potential applications for high temperature, corrosive gas sensing. The disadvantage of this sensor is that it is very vulnerable to dust and water. It requires a gas membrane film that allows the gas in and out of the fiber but not to water and dust. The fiber is currently expensive.
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