Uncooled MEMS IR Sensors for Miniaturized and Low Power Spectroscopy

A Thesis Presented
by
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to
The Department of Electrical and Computer Engineering
in partial fulfillment of the requirements
for the degree of
Master of Science
in
Electrical and Computer Engineering

Northeastern University
Boston, Massachusetts

April 2015
To my family.
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List of Acronyms

**IR** Infrared. The region of electromagnetic spectrum ranging in wavelength from the edge of the visible (~700 nm) to the edge of the microwave (~1000µm) region.

**NDIR** Non-dispersive Infrared (IR). A technique of spectroscopy which does not use dispersive elements in the optical path such as gratings or prisms. Usually, a narrow band transmission filter, corresponding to the target chemicals’s spectral signature wavelengths is used.

**MCT** Mercury Cadmium Telluride - HgCdTe. A very popular detector that spans a wide range of wavelength sensitivities. Used in almost every area of IR detection, from space telescopes to heat-seeking missiles.

**ATLM** Arbitrated Transaction Level Model. A high level communication system where the details of communication between modules are separated from the functional units comprising the computer architecture. In addition to what is provided by the Transaction Level Model (TLM), it models arbitration on a bus transaction level.

**TLM** Transaction Level Model. A high level communication system where the details of communication between modules are separated from the functional units comprising the computer architecture.

**FT-IR** Fourier Transform- Infrared. A technique of spectroscopy wherein a fourier transform of interferogram is done to obtain an IR spectrum. It is fast and avoids dispersive elements in the system set up. It can be used to obtain the emission, absorption, photoconductivity or Raman scattering of solids, liquids or gases. It is one of the standards currently for IR characterization.

**QCL** Quantum Cascade Laser. A semiconductor laser that uses quantum well heterostructures to emit electromagnetic radiation in the IR region, first demonstrated in 1994.

**G-AlN** Graphene-Aluminum Nitride. Resonator material made up of a top layer of graphene floating electrode and Aluminum Nitride, which is a piezo material.

**IPA** Isopropyl Alcohol. Also called 2-propanol. Used as a solute in this work.

**STP** Standard Temperature and Pressure. Condition where T=296 K and pressure is 1 atm.

**TCF** Temperature Coefficient of Frequency. A measure of how much shift in resonance frequency occurs for unit change in temperature. Units are in ppm/ K

**CW** Continuous Wave. Laser operation mode where the beam is constant with time and not pulsed.
Acknowledgments

I would first like to express my deepest gratitude to my advisor, Dr. Matteo Rinaldi for providing me this wonderful opportunity to work as part of his research group. Without his constant encouragement, support and guidance, this thesis would not have been possible. His enthusiasm and knowledge of MEMS and the way he approaches problems has set a standard for me to strive towards as a researcher.

I also would like to thank my research group’s members, especially Yu Hui and Zhenyun Quian, who guided me throughout the research work and helped me set up my experiments. They also taught me various concepts and instrument operation methods, that proved to be invaluable for the experiments done. Their advice and sharing of their experiences gave me an insight to what is required of researches and has set a benchmark for me to imitate.

I would also like to convey my deepest thanks to Dr. Mark F Witinski of EOS Photonics for his initial guidance on setting up an optical table and his help in letting us borrow the laser for testing. Without his help, this area of research may not even have been investigated.

I am also deeply thankful to Scott McNamara, who let me borrow chemicals from his lab at very short notice. Without his timely help, it would not have been possible to finish the thesis in time.

Finally, I would like to express my sincere gratitude to my family, for their unwavering love and support (both emotionally and technically), which helped me get through tough times over the course of this research.
Abstract of the Thesis

Uncooled MEMS IR Sensors for Miniaturized and Low Power Spectroscopy

by

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Master of Science in Electrical and Computer Engineering

Northeastern University, April 2015

Dr. Matteo Rinaldi, Adviser

While IR spectroscopy is an established technique for chemical sensing applications, practical systems are often limited by cooling requirements, limited sensitivity, large footprints, and complicated setups. In this thesis, two new IR-based spectroscopic sensors are proposed that could overcome several of these limitations. The first system is an IR spectroscope based on a G-AlN IR-sensitive resonator that can be used for both detection and concentration estimation of analyte materials - a proof-of-concept experimental demonstration is shown. It was tested using a measurement setup consisting of a CWL QCL of 5 µm wavelength as the illumination source, and a transparent liquid sample holder of 1 mm path length containing solutions of known concentration. Two liquid solutions of varying concentrations were tested: water-acetone and IPA-acetone. Comparative measurements between concentrations of 0-10 % by volume were done for both cases: the maximum detectable concentration being set by the more absorptive water and the minimum by the sensors maximum response for a 0 % concentration. The minimum detectable concentration was 0.5 % (due to noise) and the sensor is shown to be able to clearly differentiate between the two solutions, as required of spectroscopic sensors, paving way for further development to enhance sensitivity and detect extremely small concentrations.

The next part of the thesis involves a proposal for a novel, zero-power, small footprint hot exhaust plume detector for detecting vehicular exhausts from a distance. A realistic theoretical assessment of the proposed system based on a novel uncooled, temperature-compensated, bimaterial cantilever switch is done to establish the design requirements for developing the actual detector. Based on simulations for a hot CO₂ gas plume exiting a vehicle tailpipe, it is found that for a detector size of 200x200 μm², and a detection threshold of 500 nW, the detection capability ranges from 5 m to 60 m, depending upon the temperature of the exhaust plume (600 -800 K) and the size of the...
collecting optic used. Thus, it predicted that the proposed zero power detector has immense practical application possibilities, and shows great potential for further research and application in ultra-low power remote sensor systems.
Chapter 1

Introduction

The focus of this Masters Thesis is to explore the use of IR spectroscopy for sensing applications. IR spectroscopy has proved extremely useful in applications such as long distance sensing and hazardous material analysis due to IR wavelengths’ innate low scattering and absorption compared to visible light which also allows for non-contact type analysis. However, there are several key practical considerations in building IR spectroscopic systems that limit their widespread application. For example, some sensors are extremely sensitive like Mercury Cadmium Telluride - HgCdTe (MCT) but require cumbersome cooling setups to achieve it, while others like microbolometers do not require cooling, but have a much smaller sensitivity. Furthermore, all spectroscopic systems have a considerable footprint, requiring opto-mechanical setups, signal processing equipment and data analysis. This renders them useless in applications requiring long term usability such as battery-powered, remote, networked sensing systems, required by the military and environmental monitoring. Thus there is a need to develop new kinds of sensitive, uncooled sensors and ultra-low power remote sensing systems.

In this thesis, two sensor systems: one based on absorption and the other based on emission spectroscopy are demonstrated. The key contribution of the absorption spectroscopy work is the design and experimental demonstration of an IR spectroscope based on an IR-sensitized piezoelectric resonators such as Graphene-Aluminum Nitride (G-AlN). The motivation behind using resonators stems from their high sensitivity to temperature changes and ease of incorporating spectrally selective absorbers. The IR emission spectroscopy work, on the other hand, focuses on the design requirements for sensors capable of detecting gas molecules from heated sources such as vehicle exhausts. The motivation here is the development of near-zero power sensors that are 'normally-off' in the absence of analyte materials - thereby enabling near-indefinite deployment of 'in-the-field' powered sensors.
CHAPTER 1. INTRODUCTION

which are only powered-on by a positive detection of the analyte gas.

Chapter 2 provides a comprehensive background on the field of IR spectroscopy. Applications are described in Section 2.2. Fundamental concepts of IR spectrometry are described in Section 2.3. The types of IR spectroscopy, namely absorption and emission spectroscopy is described, which gives a general idea of the kind of setup used for the two types. The advantages of IR spectroscopic sensing versus other types of chemical sensing is highlighted in Section 2.4.1. A study of some popular modern spectroscopic technology is done in Section 2.4.2 both on a system level and the sensor level, with an overview of current state-of-the-art sensing methods in Section 2.4.3. Since the spectral selectivity of the sensors used in this work is an important parameter, a survey is done on the latest research on spectrally selective sensors and which type was used for the two systems presented in this thesis (see Section 2.4.3.1).

Chapter 3 deals with one of the two systems presented as part of this thesis. AlN resonators in general and graphene-AlN resonators and the motivation for using them as sensors are introduced in Section 3.1. Specific importance given to their temperature sensitivity and how these can work as effective IR sensors. Section 3.2 details the measurement setup and measurement process used to test the sensor for its utility as a spectroscopic sensor. Results for this spectroscopic system using an IR-sensitive resonator is presented in Section 3.3, with subsequent discussion on the potential applications and improvements that can be made.

Chapter 4 details the second half of the thesis, which is a proposal for a novel zero power MEMS IR gas detector. It is aimed at detecting vehicles at a distances based on the emission signature of their exhaust plume. Section 4.1 describes the motivation for developing such a low power sensor. The concept behind detecting hot gases, specifically vehicle exhaust plumes and the detection technique on which the system is based is explained in Section 4.2. Section 4.3 describes the proposed design for the detecting structure and its working. In Section 4.4, the detection system is theoretically modeled to mathematically estimate the amount of power that the detector actually receives from a hot plume at a distance, since this value is crucial for estimating the working distance range of the detector. The results of the estimations are discussed in Section 4.4.1. Section 4.5 outlines a proposed testing plan for the detectors after fabrication is complete.

Chapter 5 summarizes the work done in the thesis, with possible future plans for these two spectroscopic systems.
Chapter 2

Background

This chapter covers all the necessary background information about IR spectroscopy, from the IR region of the electromagnetic spectrum to the state-of-the art technologies in spectroscopy today.

2.1 The IR region of the electromagnetic spectrum

IR radiation was first discovered in 1800 by astronomer William Herschel by noticing the temperature increase in a thermometer on exposure to rays of sunlight beyond red light. This ushered in the previously unimagined fields of technology and applications. The IR region in the electromagnetic spectrum stretches from the edge of the visible red to the microwave region, spanning a range from ∼700 nm to around 1 mm [1] as shown in Figure 2.1. The divisions of the regions of the IR spectrum arose from the different kinds of technologies used in IR sources and detectors. Various fields of applications categorize differently; the one shown here is based on the divisions made for the different types of detectors.

Shown in Table 2.1 is the categorization of the IR spectrum based on the spectral bands of well used IR detectors according to [1]. Si based detectors are limited to 1 μm of wavelength sensitivity, whereas PbS and GaInAs detectors have a 3 μm limit. Similarly, PbSe, PtSi, InSb and MCT are used for wavelengths below 6 μm; and MCT optimized for 8-14 μm are used for wavelengths lesser than 15 μm. doped silicon sensors are used for the VLWIR region.

Different applications have been developed based on the region of the IR spectrum. One of the most prolific use of IR has been in IR data transmission. The most common of this is the humble TV remote control, which uses photodiodes as an NIR detector. Another major use, that is...
CHAPTER 2. BACKGROUND

Figure 2.1: The electromagnetic spectrum (Source [1])

<table>
<thead>
<tr>
<th>Region of IR spectrum</th>
<th>Wavelength Range (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Near infrared (NIR)</td>
<td>0.78-1</td>
</tr>
<tr>
<td>Short wavelength IR (SWIR)</td>
<td>1-3</td>
</tr>
<tr>
<td>Medium wavelength IR (MWIR)</td>
<td>3-6</td>
</tr>
<tr>
<td>Long wavelength IR (LWIR)</td>
<td>6-15</td>
</tr>
<tr>
<td>Very long wavelength IR (VLWIR)</td>
<td>15-30</td>
</tr>
<tr>
<td>Far infrared (FIR)</td>
<td>30-100</td>
</tr>
<tr>
<td>Submillimeter (SubMM)</td>
<td>100-1000</td>
</tr>
</tbody>
</table>

Table 2.1: The categories of IR radiation [1]
CHAPTER 2. BACKGROUND

declining now is the CD-ROM read/write mechanism, which IR lasers to read and write data. Other NIR devices find use in fiber optic telecommunication due to low attenuation in the fiber material that is typically SiO$_2$. Night vision goggles and cameras are also used in this region. SWIR devices are typically used for long distance communications in the 1.53-1.56 µm range. MWIR devices have applications in the military such as detecting IR signatures of aircraft and vehicles for homing missiles and bombs. The mode of detection is through thermal emissions, though the temperature of the source object has to be significantly higher than room temperature. The LWIR is the true thermal imaging region, where the sensors can detect objects that are just a little hotter than room temperature such as the human body. All the regions of the IR spectrum can be used in spectroscopic analysis. The region of the spectrum that this thesis focuses on is the MWIR region, namely at 5 µm. A wider range of applications is discussed in 2.2.

2.2 Applications of IR technology

IR technology has developed in leaps and bounds ever since its discovery two centuries ago. Nowadays, IR devices find use in almost every field, both in consumer products and cutting-edge laboratory research equipment. Some of these are highlighted below.

2.2.1 IR Thermography

IR thermography is used to image a scene to provide the temperature of every point in the scene as composite image similar to Figure 2.2. It converts the power received from a body emitting IR radiation based to its temperature using the Stefan-Boltzmann law $\frac{2}{\pi}$. This temperature is overlaid on the scene being imaged to provide an image as shown in Figure 2.2 to the user. This allows for a visualization of relative temperature over a wide area, proving invaluable to situations like heat distribution in various structures and those involving zero visibility, such as search and rescue.

2.2.2 Night Vision

Night vision is different from thermography, because it does not rely on the heat given off by objects, rather, it collects and amplifies light in the NIR region. This light consists of the ambient light like starlight or moonlight that is collected through lenses and sent to a photocathode tube converting the the photons to electrons. The electrons are amplified to a greater number through
an electrical and chemical process. The multiplied electrons are then converted to a visible image that can be seen through the eyepiece [4].

2.2.3 Hyperspectral Imaging

Hyperspectral imaging is an upgrade over ordinary spectroscopy in the fact that it can capture the complete spectral data in the scene under observation. Each pixel in the sensor contains the information for a range of IR wavelengths, proving to be an invaluable tool in remote chemical monitoring in hazardous situations. Like a thermographic camera, a hyperspectral camera can overlay the spectral data over a real image, with the only difference being the amount of information that can be visualized. These cameras, like thermographic cameras can function without needing an illumination sources. They are typically used in geological measurements and aerial surveillance. A hyperspectral image composite for a range of wavelengths is shown in Figure 2.3.

2.2.4 Astrophotography

The visible part of the spectrum is only a tiny fraction of all the radiation that exists in the universe. The human eye can only gain so much information through these wavelengths. Since a lot of the visible wavelengths are absorbed by different molecules in outer space, our knowledge of the universe was very limited. With the development of IR telescopes both on earth and space, the database of information on celestial objects and phenomena has increased multifold. Cold, normally
invisible clouds of gas glow in the IR region from the heat of nearby stars as shown in Figure 2.4. IR astrophotography has also allowed for observing the core of galaxies, normally obscured by the intervening media. Distant galaxies are moving away faster from our viewpoint, causing a redshift in their spectrum. Thus, these galaxies are more easily imaged with IR telescopes. Also, it has enabled the detection of planets surrounding stars which is advantageous because, the emission of the stars in the visible region is greater in intensity than in the IR, preventing visible observations due to the glare.

2.2.5 Meteorology

IR radiometry has been used extensively in the field of weather monitoring and prediction. Satellites equipped with IR scanners map the earth to produce thermal images, which enables meteorologists to know about various atmospheric properties over a region such as water vapor content, cloud cover, sea surface temperature, etc. They usually operate in the 10-13 µm region. High clouds are colder and show up as white, and lower clouds show up as shades of gray. The warmer land and sea show up as black. The major advantage over these IR scanners over visible ones is that they can work even on regions of the earth not facing the sun directly or in the night. A wealth of data gathered from such devices has enabled the monitoring and prediction of long term phenomena such as El Nino global warming. Figure 2.5 shows a satellite image of a hurricane over the Gulf of
CHAPTER 2. BACKGROUND

Figure 2.4: False color image of the Cygnus-X star formation region (Source: The Herschel space telescope)

Mexico.

2.2.6 IR Reflectography

IR radiation is also used to authenticate works of art. Normally, the original painter does an underdrawing on canvas first, and then paints over it. In visible light, the underdrawing is done typically by carbon black, after which the pigments are added. In visible light, the underdrawing is obscured by the pigments, since they absorb most of the light. But in IR light, the pigments are transparent, being absorbed only by the carbon black layer underneath. What results from an IR scan of the image is the underdrawing only, made by the original artist. If there are other lines in carbon black over some layers of paint, it can be detected easily, allowing art historians to decide whether a painting is the prime version or has been altered or forged.

2.2.7 Spectroscopy

Spectroscopy using IR radiation is a powerful technique for identifying and characterizing compounds or functional groups and even predict the behavior of complex molecules. It is one of the most important tools in analyzing organic molecules, which typically have regions in the IR spectrum where they strongly absorb the radiation. Every bond has characteristic absorption regions, to allow identification of molecules accurate. Spectroscopy can be done on chemicals at all phases: gas, liquid and solid. It has proved invaluable in forensic analysis, helping police to identify chemicals in evidence to pinpoint location of crime. It is also helpful in determining the blood alcohol content of
people suspected to be driving under influence of alcohol. There has been continued interest in this area and there have been more and more techniques of spectroscopy being developed over the years, turning large cumbersome machines with analog readouts to small portable devices with wireless capabilities. More details on IR spectroscopic techniques is can be found in 2.4. The following sections go into more detail about the basics of IR spectroscopy and spectroscopic techniques.

2.3 Fundamentals of IR spectroscopy

IR spectroscopy works on the premise that all molecular bonds, at any temperature emit IR radiation due to their inherent vibration at certain frequencies depending on its emissivity at that frequency of IR. The types of vibrations are briefly discussed first, followed by the important laws governing IR spectroscopy.

2.3.1 Molecular Vibrations

Molecular vibrations are broadly classified into stretching and bending vibrations. These vibrations arise due to the fact that chemical bonds are analogous to a spring with very little damping
and more than one degree of freedom. In other words, the bond angles specified in chemistry textbooks list the average bond angle; in reality, the bond angle varies in a oscillatory fashion - a result of the thermal vibrations. It is to be noted that, for a particular mode of vibration to be IR sensitive, it must correspond to a change in dipole moment in the bond, though a permanent dipole is not necessary [6]

A molecule consisting of \( n \) atoms has \( 3n \) degrees of freedom - each atom can move in the x,y or z direction. In non-linear molecules such as \( \text{H}_2\text{O} \), 3 of the \( 3n \) degrees of freedom are rotational, 3 are translational, and the rest correspond to fundamental vibrations. In linear molecules, the number of translational degrees of freedom are same, but the number of rotational degrees are 2. Therefore, non-linear molecules have \( 3n - 6 \) modes of fundamental vibrations, whereas linear molecules have \( 3n - 5 \). An example is represented in Figure 2.6 showing the various types of vibrations in a \( \text{H}_2\text{O} \) molecule. Note that this rule does not apply to functional groups such as the methyl (-CH\(_2\)).

![Figure 2.6: Fundamental modes of vibration in H\(_2\)O (Source: [7])](image)

Similarly, Figure 2.7 represents the four fundamental modes of vibrations in \( \text{CO}_2 \), which is a linear molecule. The asymmetrical stretch is of particular note because it vibrates with a wavelength of 4.25 \( \mu \text{m} \), which in wavenumber terminology is given by:

\[
\nu = \frac{10000}{\lambda} \tag{2.1}
\]

\[
= \frac{10000}{4.25} \tag{2.2}
\]

\[
= 2350 \text{ cm}^{-1} \tag{2.3}
\]

Where, \( \lambda \) is the wavelength in \( \mu \text{m} \) and \( \nu \) is the wavenumber in \( \text{cm}^{-1} \). This vibration at 2350 \( \text{cm}^{-1} \) is particularly strong and is used frequently for identifying \( \text{CO}_2 \) interference in spectroscopic analysis of other molecules.

The two bending vibrations in \( \text{CO}_2 \) are identical in space, and are called degenerate modes since they have the same frequency (666 \( \text{cm}^{-1} \)). Thus, even though \( \text{CO}_2 \) has four possible fundamental
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modes of vibration, IR absorption occurs only at 2350 cm\(^{-1}\) and 666 cm\(^{-1}\), as shown in Figure 2.8. The absorption spectrum was simulated from the HITRAN 2012 database [8].

For molecules such as H\(_2\), N\(_2\) and Cl\(_2\), there is no change in dipole moment due to vibrations and are therefore IR inactive. Typically, asymmetric molecules absorb in the IR region and greater the change in dipole moment, greater the absorption peak in the IR spectrum for that molecule; and the more complex a molecule, greater is the number of absorption peaks in the IR spectrum. Weaker absorption peaks that correspond to overtones (usually at double fundamental frequency) and combinations of fundamental modes are also sometimes present in the absorption spectra. Stretching vibrations, can be modeled as a spring with two masses on the ends as shown in Figure 2.9. Thus, it can be described using Hooke’s Law for simple harmonic oscillators.

According to the law, the vibration frequency is dependent on the mass and spring constant by the relation:

\[
\nu = \frac{1}{2\pi} \sqrt{\frac{k}{m}}
\]  

(2.4)

where, \(k\) is the spring constant, \(\nu\) is the vibration frequency and \(m\) is the effective mass given by \(m = m_1m_2/(m_1 + m_2)\). But in the case of true simple harmonic oscillators, the energy of the spring is given by: \(E = h\nu = \frac{1}{2}kx^2\), where \(x\) is the displacement of the spring. If it were the case for the molecular model, then the bond would absorb radiation of all frequencies. In reality, the vibrational motion of molecules is quantized, which implies that only certain energies of vibrations
CHAPTER 2. BACKGROUND

Figure 2.8: IR absorption spectrum of a thin layer CO$_2$

Figure 2.9: Molecular spring model (Source: [7])
are allowed. The allowed energies is given by:

\[ E = \left( n + \frac{1}{2} \right) h\nu \]  

(2.5)

where, \( h \) is Planck’s constant and \( n \) is the quantum number (0,1,2,3...). Accordingly, only transitions to the next level is allowed, which is \( h\nu \) more than the previous levels. Sometimes jumps to energies greater than \( h\nu \): \( 2h\nu \) or \( 3h\nu \) are observed, and these frequencies show up in the IR spectrum as “overtones”, but have lower intensity. Shown in Figure 2.10 is the comparison between a normal spring and a molecular one.

![Energy-displacement diagram for molecular springs](Source: [7])

Ideally, these equations would hold true, if not for the nature of molecular bonds namely, the bonds can come apart and the bonds cannot be compressed beyond a certain distance. The energy reaches a maximum with interatomic distance and then decrease with the energy levels becoming more closely spaced for greater distances. Thus, as shown in Figure 2.11, the quantum energy of overtones that was predicted in the previous equation would higher than the actual energy.

Apart from overtone bands, other bands are observed in the IR spectrum. *Combination* bands occur when two fundamental bands absorb energies simultaneously at two frequencies \( \nu_1 \) and \( \nu_2 \), causing a band to appear at \( \nu_1 + \nu_2 \). *Fermi resonance* occurs when overtone or combination band occurs at the same frequency or close to a fundamental band. This causes the appearance of two peaks close to each other, with the split in the middle occurring at the expected frequency. *Coupling* occurs when the vibration of one bond is transmitted to adjacent bonds in a molecule, causing modes
of vibrations that are due to those other bonds. For this to happen, the bonds must have similar frequencies and must be part of the same molecule [9]. Vibration-rotation bands are observed in gaseous species and are visible as closely spaced components in high resolution spectra. This arises due to excitation of rotation modes by a vibration mode transition. This causes the overall absorption band to be split into P, Q and R bands depending on the rotational quantum number. The distance between the closely spaced lines can be used to determine bond length [9].

2.3.2 Blackbody Radiation

All objects emit electromagnetic radiation from to the vibrational transitions of molecules and subatomic due to their temperature. The higher the temperature, the faster is the vibration and total radiated energy. Therefore all objects continuously emit radiation across the wavelengths of the spectrum, with the intensity at each wavelength dependent upon its temperature and emissivity which is a function of the wavelength. A blackbody is an object that absorbs all radiation and emits all radiation. Thus, the energy emitted by a blackbody at a given temperature is the maximum theoretical limit at that temperature. Thus, approximations of blackbodies are used as calibrators for instruments measuring radiation. Other sources of radiation emit in fashion similar to a wavelength-filtered blackbody: at certain wavelengths, the energy emitted is lower than a corresponding ideal blackbody.


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This characteristic is defined by the wavelength-dependent emissivity. An ideal blackbody can be described by the Planck equation:

\[
L(\lambda, T) = \frac{2hc^2}{\lambda^5} \left[ \exp \left( \frac{hc}{\lambda kT} \right) - 1 \right]^{-1} [W/(cm^2 sr \mu m)] \tag{2.6}
\]

\[
M(\lambda, T) = \pi L \ [W/(cm^2 \mu m)] \tag{2.7}
\]

where, \( L \) is the Radiance of the blackbody, in other words, it is the power emitted by the surface of a body per unit area per unit solid angle at a particular wavelength; \( T \) is the temperature of the body, \( c \) is the speed of light, \( \lambda \) is the wavelength and \( k \) is Boltzmann’s constant. \( M \) is the radiant exitance or the power exiting the surface per unit area. Figure 2.12 shows the spectral distribution of the radiant power of blackbodies at various temperatures simulated in MATLAB. The higher the the temperature, the higher is the total power radiated and the smaller is the wavelength of the peak of the curve. This is the reason why less hot objects glow a dull red and hotter objects glow brighter and less red. In fact the hottest stars in the universe glow blue in contrast to a medium hot star like the sun which glows white. Thus, if an IR sensor is to be used as a thermal sensor, its sensitivity should encompass the wavelengths of radiation around the peak wavelength of the object radiating. For example, an IR thermal sensor for a furnace operating at 2000 K would have maximum efficiency if its peak sensitivity was centered around 2.5 µm.

![Figure 2.12: Spectral radiance of ideal blackbodies](image)

Figure 2.12: Spectral radiance of ideal blackbodies
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The actual spectral radiance of a non-blackbody source is determined by its emissivity, $\epsilon(\lambda, T)$, which is given by:

$$
\epsilon(\lambda, T) = \frac{L(\lambda, T)_{\text{source}}}{L(\lambda, T)_{\text{blackbody}}} = \frac{M(\lambda, T)_{\text{source}}}{M(\lambda, T)_{\text{blackbody}}} \quad (2.8)
$$

$\epsilon$ is a dimensionless number and is equal to 1 for an ideal blackbody. A “graybody” is one whose emissivity is independent of wavelength and the total radiant exitance for it is given by:

$$
M_{\text{graybody}} = \epsilon \sigma T^4 \quad (2.9)
$$

where $\sigma$ is the Stefan-Boltzmann constant. When radiation is incident on a surface, a fraction of the energy is absorbed, reflected or transmitted. The following relates the three:

$$
\alpha + r + t = 1 \quad (2.10)
$$

where $\alpha$ is the absorptance, $r$ is reflectance and $t$ is the transmittance. According to Kirchoff’s law, for a given temperature, the ratio of the integrated emissivity to the integrated absorptance is constant for all materials and is equal to the radiant exitance of a blackbody at that temperature:

$$
\frac{M(\lambda, T)_{\text{source}}}{\alpha} = M(\lambda, T)_{\text{blackbody}} \quad (2.11)
$$

$$
\frac{\epsilon \sigma T^4}{\alpha} = \sigma T^4 \quad (2.12)
$$

Thus, $\epsilon = \alpha \quad (2.13)$

For opaque objects, $t = 0$ and so $\alpha + r = 1$ and $\epsilon = 1 - r$. Figure 2.13 shows the difference in spectral radiance of graybodies and non-graybodies (selective radiators/absorbers) versus an ideal blackbody. An example of a selective radiator is glass, which transmits extremely well at some wavelengths including the visible region, but is almost completely absorbent at some IR regions.

2.3.3 Absorption and Emission Spectroscopy

The phenomenon of selective absorption and emission is what spectroscopy is based on. Consider Figure 2.13. If a blackbody illuminates a selective absorber that is selectively transparent, the observer on the other side of the absorber will see a spectrum of the curve that looks like the one shown on the right extreme of the figure. An explanation is demonstrated in the schematic shown in Figure 2.14. A close-to-ideal blackbody source with a known temperature and spectral radiance,
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Figure 2.13: Spectral radiance of different objects

acts as a source of radiation for the sample - in this case CO₂ - which absorbs some radiation and transmits some radiation. The transmittance or inverse of absorptance of the gas for the range of wavelengths is shown in the middle graph and this acts as a filter for the incident radiation.

The result as seen from the other side is a spectrum as shown in the third graph. These were simulated in MATLAB using the equations of radiative transfer. The equation of radiative transfer allows for converting the measured signal to a variable of interest such as concentration or temperature of the sample. The radiative transfer equation is written as:

\[ \frac{dL_\nu}{dz} = \varepsilon_\nu - k_\nu(z) L_\nu \]  

(2.14)

where, \( L_\nu \) is the spectral radiance of the source, \( z \) is the distance along axis of measurement, \( \varepsilon_\nu \) is the emission coefficient and \( k_\nu \) is the absorption coefficient. Note that, from here on, all relevant quantities will be expressed in terms of wavenumber (cm\(^{-1}\)) instead of wavelength (\(\mu m\)) for convenience. Subscript \( \nu \) implies wavenumber dependence. On solving the equation assuming there is only emission and absorption in the radiative transfer and no scattering, fluorescence or phosphorescence effects we get:

\[ L_\nu(l) = L_\nu(0)e^{-k_\nu l} + S_\nu \left[ 1 - e^{-k_\nu l} \right] \]  

(2.15)
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Figure 2.14: Schematic for the spectral measurement of an absorptive gas

where, \( l \) is the path length and \( S_{\nu} \) is the source (illumination) function given by \( S_{\nu} \equiv \varepsilon / k_{\nu} \).

For absorption spectroscopy, wherein self emission of the sample is negligible, only the first part of the equation is taken into account. This form of the equation is called Beer-Lambert’s law and describes the absorption as a function of path length \( l \) through the absorbing medium.

\[
L_{\nu}(l) = L_{\nu}(0)e^{k_{\nu}l} \quad (2.16)
\]

\( L(0) \) is the incident spectral radiance before it enters the sample and \( L(l) \) is the spectral radiance transmitted by the gas/absorbing media. This equation was used to simulate the graphs in Figure 2.14. The absorption coefficient \( k_{\nu} \) was calculated from the line strength data for \( \text{CO}_2 \), which was obtained from the HITRAN database \[8\] using the methods described in \[10\] using the formula:

\[
k_{\nu} = P\chi_{m}S_{\text{line}}(\nu,T)f_{\nu \rightarrow \nu_0} \quad (2.17)
\]

where, \( P \) is the total pressure of the gas sample, \( \chi_{m} \) is the mole fraction of the gas species, \( S_{\text{line}}(\nu,T) \) [\( \text{cm}^{-2}/\text{atm} \)] is the temperature-corrected line strength data calculated as:

\[
S_{\text{line}}(\nu,T) = S_{\text{line}}(\nu,T_{\text{ref}}) \times 2.68676 \times 10^{19} \times \frac{296}{T} \quad (2.18)
\]

where \( 2.68676 \times 10^{19} \) is the lochschmidt’s constant [\( \text{molecules/cm}^3 \text{ atm} \)] and \( S_{\text{line}}(\nu,T_{\text{ref}}) \) is the the line strength [\( \text{cm}^{-1}/\text{molecules cm}^{-2} \)] at STP (296 K, 1 atm). This line strength data is a molecule-dependent property and indicates how strongly a molecule can absorb or emit radiation. \( f_{\nu \rightarrow \nu_0} \) models line broadening effects due to the dynamics of the molecules, such as motion of molecules that causes a Doppler shift in the observed frequency, and due to collisions between...
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molecules. The ‘Voigt’ lineshape models this function. More information about this can be found in [10]

The first part of this thesis is based on absorption spectroscopy wherein spectroscopic measurement of a liquid solution is done (the gas in Figure 2.14 is replaced by a liquid sample of known path length). The power transmitted through the liquid solution can be determined by another form of the Beer-Lambert’s law, which is used for finding the concentration-dependent absorption:

\[ L_\nu(l) = L_\nu(0) \times e^{-a_l c} \]  

(2.19)

where, \( a \) is the molar absorptivity of the solute (in \( L \text{ mol}^{-1} \text{ cm}^{-1} \)) and \( c \) is the concentration of the solute (in \( \text{mol} \text{ L}^{-1} \)). Thus, in absorption spectroscopy, the concentration can be estimated by measuring the power received by the sensor and converted to the concentration. In the work presented, a solution of water in acetone and IPA in acetone are tested separately for different concentrations, and it is seen if the response obtained matches the expected trends. Since water has a higher absorptivity [11], compared to chemicals like Isopropyl Alcohol (IPA) and acetone, it is expected to show a steeper variation in response for different concentrations compared to the IPA-acetone solutions. A graph comparing the expected trends of both these solutions for different concentrations is shown in Figure 2.15. It was calculated using the above Beer-Lambert’s equations for concentrations ranging from 0 % to 10 % by volume. The molar absorptivity is taken as arbitrary constants proportional to the absorbances obtained from Figure 3.7. It is noted that molarity of these two solutes will be different and it has also been taken into account using the concepts of dilution and incorporated as the values for the concentration \( c \).

In case of emission spectroscopy, where there is no source behind the sample and the measurement is done solely using the radiation emitted by the sample itself, the second part of equation 2.15 is used:

\[ L_\nu(l) = S_\nu \left[ 1 - e^{-k_\nu l} \right] \]  

(2.20)

According to Kirchhoff’s law, the source function \( S_\nu \) becomes the blackbody function \( B_\nu \) in case of emission [10]. Therefore,

\[ L_\nu(l) = B_\nu \left[ 1 - e^{-k_\nu l} \right] \]  

(2.21)

The emission spectrum for a hot gas such as CO\(_2\) is shown in a simulated plot in Figure 2.16 with a comparison to an equally hot blackbody. The second part of the research done as part of the thesis
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Figure 2.15: Graph showing comparison of expected responses of the sensor for the absorption system

Figure 2.16: Emission spectra of CO$_2$
involves the manipulation of emission spectra of hot gases to propose a new type of passive gas detector.

Thus, the concept of spectroscopy in general is a powerful tool in analyzing chemical compounds and has been manipulated in different ways for different applications in spectroscopy. Some of the popular spectroscopic methods have been discussed in 2.4.

2.4 IR spectroscopy - An overview of current technology

A plethora of non-contact sensing techniques have been developed over the years since the pioneering work by Fraunhofer using dispersive spectroscopy [12]. A few of the modern ones that have been developed to work in the Infrared region include Dispersive spectroscopy, Fourier Transform- Infrared (FT-IR), Time-resolved spectroscopy, Tunable laser spectroscopy and Non-dispersive IR (NDIR) spectroscopy. This section describes some of the recent technologies (sensors and systems) of spectroscopy that warrant mention, highlighting the advantages and disadvantages of each.

2.4.1 Spectroscopic vs. other methods of chemical sensing

It should be noted that it is possible to analyze chemicals using a variety of different methods such as mass-spectrometry, chromatography, calorimetry, thermogravimetric analyses, etc. In all of those techniques, the sample has to be in contact or close proximity to the sensor and the measurement process itself may be destructive to the sample. There are some other less complex systems for gas sensing, with a narrow range of chemicals that can be detected. These are typically sensitive to the chemical being detected, as they rely on a selective absorption layer as the main sensing component. Conductometric, Electrochemical and catalytic bead are the different types of contact type sensors worthy of mention. These sensors, which will be referred to as contact-type sensors, suffer from the basic flaw that they have to physically come in contact with the medium being sensed. This is a crippling factor when it comes to applications such as remote sensing and situations where it is simply not possible to come in contact with the sample. Thus, these techniques cannot be used remotely in inaccessible areas. Such applications include detection of exhaust gas plumes at a distance, from a few mm to billions of km such as used in astronomical observations. Also, there is no possibility of estimating the spatial distribution of the chemical, since the measurements are made at a point in space, unlike technologies such as hyperspectral cameras. In fact, what we know now
of the cosmos was only possible due to advances in non-contact measurement techniques, namely spectroscopy.

2.4.2 Modern Spectroscopic Systems

Dispersive IR spectroscopy is one of the most prolific of all techniques and has been a tried and tested method ever since Isaac Newton discovered the spectrum by passing white light through a prism. Here, a broadband IR source is split into its component wavelengths by a prism or grating and each of these wavelengths is sent through the gas sample. A broad wavelength-sensitive sensor records the heat absorbed for each wavelength. This produces the well-known spectra of a particular gas. As expected, these are quite bulky owing to the optical components such as the grating, slit and optical components. The beam has to be split and sent through two paths, with one of the paths being the reference. This adds losses to beam power and ultimately results in lesser sensitivity. It is also a slow process requiring sampling across the entire range to produce the spectrum. Modern laboratories are increasingly shifting to other techniques of IR spectroscopy since they provide better performance [13].

FT-IR spectroscopy is by far the most popular IR spectroscopy instrument currently in use, trumping the dispersive technique in almost all the criteria. The FT-IR consists of a less complex system containing just two mirrors for a moving mirror interferometer and one sample chamber and produces an interferogram that contains information from each wavelength of light being measured. Every half cycle of the moving mirror in the system spans the entire range of wavelengths, thereby allowing spectral calculations at a fraction of the time that it would take for a dispersive instrument. Since there are less optical components and no slits between source and detector, there is lesser loss in beam energy, increasing sensitivity and allowing for high resolution spectra. Using an inbuilt laser wavelength calibration system, FT-IR spectrosopes do not drift their wavelength reference position over time and have a high degree of precision for measurements. One disadvantage that is becoming less of an issue is the external digital signal processing requirement to generate the spectra from the interferogram. Due to the lesser complexity of its design, there are extremely compact FT-IR spectrosopes that are currently available, rivaling the performance of large dispersive instruments [14]. For simpler applications like single chemical species detection or estimation, it is a bit overkill, as a complete spectrum is not needed.

Tunable laser spectroscopy has entered the limelight with the introduction of the tunable Quantum Cascade Laser (QCL). This is a relatively recent technique that replaces the source and
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diffraction grating of dispersive IR spectrometers with a narrow band laser beam whose central wavelength can be tuned over a small range. Having an array of such tunable lasers enables an uninterrupted high resolution scan of wavelengths over a large wavelength range. Since the source is a collimated laser, the beam can be sent through very small volume cells as well as long distance open paths. It is claimed that this system matches and outperforms FT-IR instruments in certain cases [15]. A major advantage of this technique is the complete lack of moving components, with all control being done by chip scale electronics. This implies extreme portability as evidenced in [15, 16] and the possibility of Lab-on-chip spectroscopy. Currently, the fabrication of these lasers is still expensive and has limited dissemination through research labs.

The technique that is closest to the two methods presented here is the NDIR spectroscopic technique. NDIR works on the premise that the characteristic signature of the gas to be sensed is already known. It avoids the scanning setup of all the above techniques by making the sensor sensitive to only the narrow wavelength ranges where the absorption peaks for the gas occurs. NDIR spectroscopes achieve this by placing a narrow band transmission filter in front of the relatively wide band sensing element. The sensor itself may be a wide band one, which would reduce the noise performance. Due to its simplicity, NDIR technique is used more commonly than the dispersive technique. It finds most use in industrial process control and continuous monitoring of gases such as carbon monoxide. In the experimental work of absorption-based spectroscopy presented in this thesis, the wavelength selectivity is ensured by a non-tunable QCL and an absorptive sensing layer that is sensitive to wavelengths around the QCL’s wavelength, removing the need for a transmission filter and improving noise performance. Thus, this system could be called a NDIR non-tunable laser spectroscope. In the other method that is based on emission spectroscopy, there is only the bare minimum components in the system, namely the sensors. All the wavelength selectivity and signal processing is done passively on the sensor itself, making it an extremely simplistic system for gas detection, unlike the methods described above.

2.4.3 IR Sensor Technology for Spectroscopes - The State-of-the-Art

All of the spectroscopic systems mentioned previously require some form of IR sensors, which ultimately determine the wavelength range and sensitivity. A few of the popular ones are discussed here.

Different types of IR sensors are used for different applications in IR sensing. High sensitivity applications, such as FT-IR spectroscopy and IR astrophotography in both ground and
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space-based observatories (e.g. The Hubble Space Telescope), use high-performance cooled detectors that have a wide-band flat absorption characteristic namely, MCT (Mercury Cadmium Telluride) [12, 17]. It is a semiconductor that works as photoconductive detector by absorbing photons of required energy to send an electron from the valence band to the conduction band. To reduce noise due to thermally excited carriers, it has to be cooled sometimes to the boiling point of nitrogen, thereby making it quite bulky and uneconomical. Nevertheless it is one of the most versatile types and is used in a wide range of military applications such as LWIR (long wavelength IR) imaging and heat-seeking missiles. A competitor is the photodiode type sensor made of GaInAs (Gallium Indium Arsenide) whose spectral range is smaller. Other sensors are also currently available with good performances such as microbolometers that do not require any cooling and are increasingly being used in portable IR imaging. Microbolometers work by converting the absorbed IR to temperature increase, which can be sensed electrically as say, a resistance change. Microbolometers in general are plagued by lower sensitivities and higher thermal time constants compared to the other types, though progress is being made. The major advantage of this type of sensor not needing external cooling is very appealing and holds promise for development into imaging and hyperspectral applications.

Another popular method of IR detection in imaging applications has been the utilization of bimetallic cantilevers. Since the proposed zero-power sensor for exhaust gas detection is based on such a mechanism (see 4), it warrants comparison with current research on thermomechanical cantilever technology. There has been a strong effort in developing thermomechanical sensors, mainly to use in focal plane arrays for imaging. Thermomechanical sensors are much cheaper than corresponding photon sensors [18] and typically do not need cooling. In all the recent developments, sensors are used for imaging and incorporate optical readouts where a laser reflects off the deformed switch’s surface to estimate intensity of the absorbed IR radiation. Capacitive readouts have been researched, but the fabrication was too challenging and the result did not offer great advantages compared to optical readout methods [18]. Oak Ridge National Laboratory has developed a SiOx-Al bi-material based microcantilever with optical readout using only three lithography steps [19]. Agiltron Inc. has developed an optical readout, high frame-rate FPA camera with tunable sensitivity that has a novel bi-material temperature-compensated sensor structure in each pixel [20]. The design for zero power gas detector presented in this thesis will use a modified version of this structure. Another group has used a different arch type bi-material cantilever design which uses a high CTE polymer to achieve actuation[21]. This type is a potential candidate for use in the proposed system in subsequent phases of development since it too incorporates a ambient compensation system.

In summary, to the best of knowledge, there are no truly passive, wavelength-selective
thermomechanical switch-based gas detection systems available today; all the current methods involve an optical readout mechanism and are not particularly suitable for the zero power/passive aim and are not directed towards gas detection. In [22], a very similar approach has been done with respect to the sensing layer and actuation mechanism, but it still uses optical readout, with further aims to use as an imager. The zero power sensor design, on the other hand has no external readouts or components apart from a lens or mirror for focusing the IR. It will use a similar switch structure as [23] due to the inbuilt stress and temperature compensation mechanism.

Since the sensitivity of the sensor is directly dependent on the absorption efficiency or absorptance of the sensing layer, a major part of research related to improving performance has been to maximize the radiation to signal conversion efficiency. This has produced a variety of clever techniques to confine the incident light in the absorbing layer or making the absorbing layer almost 100% absorptive. In both of the works presented here, the absorption layer is a central part of the sensors and is manipulated to absorb the required wavelengths.

2.4.3.1 Spectrally Selective Sensors

If the application requirement calls for identification/analysis of a specific chemical or group of chemicals, a widely selective absorber can be detrimental since it absorbs many wavelengths that are not needed. Since each chemical species has its own characteristic absorption lines (its IR signature), a sensor needs to be selective to only those wavelengths. The novel-zero power sensor, proposed as a part of the thesis uses such a spectrally selective absorber.

As far as selectivity of wavelength goes, the absorption layer plays a major part in the function. There has been a strong research effort in developing highly absorptive layers. Loosely classified into material dependent absorbers and pattern based absorbers, the former depends on the material properties to achieve selective IR absorption, whereas the latter uses patterned structures [24], though there is frequent overlap between the two. One of the structurally patterned detectors that has been developed is the SiC grating type absorber. It consists of a grating made of grooves etched in the material at periodic intervals and the absorption was found to be dependent on the period of these grooves in the grating, which the authors have attributed to surface standing wave modes generated in the grating [25]. It is seen in [26] that the emissivity or absorptance is wavelength and polar angle dependent. This type of absorber would not be optimal for use in in spectroscopy, as it is angle dependent and would require careful alignment for the sensor to correct angle.

Artificially microfabricated 2D and 3D photonic crystals have also been used to achieve
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spectrally narrow absorptions [27]. Tungsten was used in reference to fabricate a 3D photonic crystal to achieve a complete photonic bandgap of 8 and 20 μm. The photonic bandgap causes almost complete reflection over a broad range of wavelength and angle. The peak absorption at the 6 μm region is found to be almost 50% larger than that of flat tungsten and this was found to be due to increased interaction of the EM fields with the metal due to lowered group velocities at the band edge [28]. The absorption peaks, though quite narrow, still are relatively broadband below a certain absorption, and this would act as noise sources if used in the zero power sensor. The structures are also more difficult to fabricate compared to other techniques due to the 3D criss-crossed multi-layer structure.

One of the most significant research efforts on developing IR absorbers has been the development of metamaterial and plasmonic absorbers whose fabrication is relatively simple and whose wavelength selectivity can be tuned by the pattern of a single layer and the thickness of another. It is made up of a top layer of micron sized metallic 2D structures, smaller than the detected wavelengths, a middle layer of thin dielectric and a bottom back-reflecting metallic layer. The wavelength selectivity can be adjusted by adjusting the size and spacing of the top layer structures and the thickness of the dielectric. Multiple wavelength selectivity can be incorporated on the same sensor based on the pattern, as shown in Figure 2.17

![Absorption characteristics of metamaterial](Source:[29])

Figure 2.17: Absorption characteristics of metamaterial (Source:[29])
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Our own research group has demonstrated a metamaterial-coated resonator [30], which demonstrates the potential to have quite narrow and well defined peaks with a much higher absorbance than in the troughs. This allows for reduced noise from non-targeted wavelength sources, allowing for decreased false alarm/measurement possibilities. It is also relatively insensitive to incident angle of the IR rays. Figure 2.18 shows a rendering of a metamaterial-coated AlN resonator developed by our research group in [30]. Since the structure is not very complex, it is relatively simple to model in finite element analysis software to predict the absorbance spectrum with good certainty and fine tune required parameters before fabrication. The absorbance mechanism stems from the induction of antiparallel currents at the top and bottom layer causing confinement of the field within the dielectric at the resonant frequency. The ohmic losses in the top and bottom layer, coupled with losses in the dielectric medium causes efficient absorption. This particular type of absorber is incorporated in the proposed emission-based gas detector as described in 4.

Figure 2.18: Schematic rendering of a metamaterial-coated resonator (not to scale)

Initially, a metamaterial-coated resonator was the device of choice for the absorption-based work as well in this thesis, but the device could not be tested due to damage when wire bonding. Therefore another device with a weak Fabry-Perot absorbing cavity was tested as the sensor for the
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set up, since it was a test of concept and sensor level performance was not a priority as long as it was sensitive to IR in the desired wavelength. Fabry-Perot cavity coupled structures are the most classic type of efficient absorbers, whose absorption peaks depend on the distance between two reflective surfaces in the absorbing structure and angle of incidence [24]. Coupling this with narrow band filters would allow these types to work well as selective absorbers. One example of Fabry-Perot optically coupled devices is demonstrated in [31]. It uses a coupled absorption cavity which can be tuned electrostatically to change the distance between the reflecting surfaces of the cavity which in turn tunes the wavelength sensitivity. The device used for this thesis has a non-tunable Fabry-Perot cavity consisting of a dielectric layer of aluminum nitride sandwiched between an optically transparent graphene layer on top and a fully reflective platinum layer on the bottom. The graphene layer also acts as a floating electrode for the piezoelectric resonator while the bottom platinum layer also acts as the resonator’s electrodes. A schematic of the device is shown in Figure 2.19 with the mechanism of absorption. When the incident lights strikes the graphene layer, part of it is reflected and part is transmitted. It undergoes refraction in the dielectric (AlN) layer and reflects completely off the bottom platinum layer and again comes up to the graphene layer. If the wavelength for a particular angle of incidence is just right, the reflected light from the top layer and bottom layer destructively interfere (shown in red and blue) and there is no light exiting the graphene layer. This causes the light waves to remain confined in the dielectric until it is absorbed by it. Thus, intuitively assessing the mechanism, the interference occurs due to the phase difference introduced upon incidence, refraction and reflection, which in turn depends on angle of incidence, and the phase difference due to the decreased velocity in the dielectric. The fraction of light that is confined within the lossy dielectric also depends on the reflectivity of the top layer. Graphene, being a somewhat bad reflector, makes

![Figure 2.19: Fabry-Perot absorption mechanism in a G-AlN resonator](image)

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the cavity a weak absorber, but enough for the purposes of preliminary testing of such resonators for spectroscopic sensors. Graphene affords other desirable properties as described in 3, which if optimized for IR sensitivity could make it an extremely effective device.
Chapter 3

G-AlN Resonators as IR Sensors

This section consists of a brief introduction to the AlN resonators used in this work and the motivation to use them as IR spectroscopic sensors.

3.1 Aluminum Nitride Nanoplate Resonators

Aluminum Nitride is a popular wide band semiconductor that is widely used in the electronics industry. Its unique properties make it a material of choice for microelectronic applications. Its first applications stemmed from its high electrical resistance and relatively high thermal conductivity. Its piezoelectric properties have also been tapped for high frequency applications. Currently, it finds use in RF filters in mobile phones as thin film bulk acoustic resonators (FBAR). Other piezoelectric materials such as PZT (Lead Zirconium Titanate) and ZnO are also common piezoelectric materials, but AlN has the advantage over them because of its low RF and acoustic losses, high melting point, high acoustic velocity, low TEC (thermal expansion coefficient), GHz capability and full compatibility with CMOS processes. The latter is an important factor in AlN-enabled RF microchips that are in widespread use today [32].

Its properties arise mainly due to its crystalline structure. The Wurtzite phase structure is the one that is used as a piezo material. Piezoelectricity is the phenomenon that is characterized by the production of an electric field on application of mechanical force and vice versa. This occurs due to a small displacement of atoms in the lattice, which causes a change in the direction and moment of dipoles. These dipole moments as a sum, produce a net dipole moment that appears as an electric field across the crystal. In non-piezoelectric crystals, the individual dipoles cancel each
other’s effect resulting in a net zero electric field. Mathematically this is represented as:

\[ D = \varepsilon E \]  
\[ \Rightarrow D_i = \varepsilon_{ij} E_j \] 

where \( D \) is the electric charge density displacement, \( \varepsilon \) is called the permittivity matrix of the piezoelectric crystal and \( E \) is the applied electric field. Hooke’s law for mechanical stress and strain is given by:

\[ S = sT \]  
\[ \Rightarrow S_{ij} = s_{ijkl} T_{kl} \] 

These equations may be written as coupled equations in the strain-charge form:

\[ S = sT + d^i E \]  
\[ \Rightarrow S_{ij} = s_{ijkl} T_{kl} + d_{kij} E_k \] 

\[ D = d^i T + \varepsilon E \]  
\[ \Rightarrow D_i = d_{ijk} T_{jk} + \varepsilon_{ij} E_j \] 

for AlN crystals and similar piezoelectric crystals, the complete form of the above equations are as follows:

\[
\begin{bmatrix}
S_1 \\
S_2 \\
S_3 \\
S_4 \\
S_5 \\
S_6
\end{bmatrix} =
\begin{bmatrix}
0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 2(s_{11} - s_{12}) \\
0 & 0 & 0 & 0 & 0 & 0
\end{bmatrix}
\begin{bmatrix}
T_1 \\
T_2 \\
T_3 \\
T_4 \\
T_5 \\
T_6
\end{bmatrix} +
\begin{bmatrix}
0 & 0 & d_{31} \\
0 & 0 & d_{32} \\
0 & 0 & d_{33} \\
0 & d_{24} & 0 \\
d_{15} & 0 & 0 \\
0 & 0 & 0
\end{bmatrix}
\begin{bmatrix}
E_1 \\
E_2 \\
E_3
\end{bmatrix} \]  
\[ (3.9) \] 

\[
\begin{bmatrix}
D_1 \\
D_2 \\
D_3
\end{bmatrix} =
\begin{bmatrix}
0 & 0 & 0 & 0 & d_{15} & 0 \\
0 & 0 & 0 & d_{24} & 0 & 0 \\
d_{31} & d_{32} & d_{33} & 0 & 0 & 0
\end{bmatrix}
\begin{bmatrix}
T_1 \\
T_2 \\
T_3 \\
T_4 \\
T_5 \\
T_6
\end{bmatrix} +
\begin{bmatrix}
\varepsilon_{11} & 0 & 0 \\
0 & \varepsilon_{22} & 0 \\
0 & 0 & \varepsilon_{33}
\end{bmatrix}
\begin{bmatrix}
E_1 \\
E_2 \\
E_3
\end{bmatrix} \]  
\[ (3.10) \]
CHAPTER 3. G-ALN RESONATORS AS IR SENSORS

where \( d \) is the direct piezoelectric coefficient and \( d^t \) is converse piezoelectric coefficient matrix. \( d_{ij} \) is of interest because represents the ratio of the strain applied in a particular axis and the corresponding electric field generated in another axis and vice versa, when there are no other strains present. The specific values for \( i, j \) that are exploited depend upon the material and the application. For example, \( d_{33} \) is used in thin film bulk acoustic resonators. AlN, which is the material of choice for the resonator used in this work, is utilized by exploiting the \( d_{31} \) coefficient. A schematic of the orientations of the axis helps to clarify the axial modes involved. The crystallographic axes show correspond to a bihexagonal crystal and X and Y are separated by 120°. As shown in Figure 3.1, the vibrations are excited in the \( d_{31} \) direction by applying an electric field in the thickness direction. This mode of vibrations are called contour mode/lamb wave vibrations. AC electric fields, therefore cause the crystal structure to resonate at a particular frequency laterally, depending on the physical and mechanical properties of the structure employed.

![Figure 3.1: Schematic of \( d_{31} \) vibration excitation by electric field along thickness](image)

The unit structure of the resonator used in this work is shown in Figure 3.2a. The electric field applied across the electrodes (yellow) oscillates across the thickness of the resonator, causing lateral expansion and contraction of the AlN (shown in green). Depending on the effective density (\( \rho_{eff} \)), effective Young’s Modulus (\( E_{eff} \)) of the three layers of the resonator structure and width of
CHAPTER 3. G-ALN RESONATORS AS IR SENSORS

the electrode, the resonant frequency \( f_r \) is determined by the following relation:

\[
f_r = \frac{1}{2W} \sqrt{\frac{E_{eff}}{\rho_{eff}}}
\]

(3.11)

Figure 3.2: Unit structure (a) and the actual symmetric structure used (b)

The actual structure made of repeating units of these cells is shown in Figure 3.2b. It consists of alternating electrodes arranged in a symmetrical pattern, all lying on the same plane. On the bottom side of the structure is a floating electrode that reflects the electric field towards the opposite electrode; without it, the electric field will not traverse the entire thickness of the AlN. This form of electrode patterning is much easier to fabricate and allows for using the floating electrode side as a sensing layer, which is what is done in the device used for the absorption-based system. The thickness of the AlN, number of fingers \( n \) and length determine the effective impedance of the structure by affecting the motional resistance \( R_m \) and capacitance \( C_0 \).

\[
R_m \propto \frac{T}{nL}
\]

(3.12)

\[
C_0 \propto \frac{nLW}{T}
\]

(3.13)

A resonator can be represented as an equivalent lumped electric circuit, called a Butterworth-Van-Dyke model. As shown in Figure 3.3 it consists of an RLC circuit with two branches - a static branch that consists of capacitances associated with the geometric capacitance of the structure and parasitic capacitances, and a motional branch associated with the mechanical resonance due to the vibration. \( R_s \) and \( R_0 \) represent series and parallel resistances due to parasitic effects due to contact resistance and leakage through substrate. The series and parallel branches cause two resonances to occur that are called series and parallel resonance, where the admittance is purely resistive and the
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reactances of the capacitance and inductances cancel each other. These resonant frequencies change with changing temperatures, allowing the resonator to be manipulated to work as a thermal sensor.

\[
TCF = \frac{1}{f} \frac{\partial f}{\partial T} = -\alpha_1 + \frac{1}{2} \frac{\partial \ln E}{\partial T} + \frac{1}{2}(2\alpha_1 + \alpha_3)
\] (3.14)

where \(\alpha_1\) and \(\alpha_3\) are the linear coefficients of thermal expansion for the piezo material (AlN) in the plane of the film and out of the plane of the film respectively. \(\frac{\partial \ln E}{\partial T}\) is the variation of the in-plane modulus of elasticity with temperature. The largest contribution to TCF is from this term and is typically around 37 ppm/°C as described in [33, 34]. Experimentally, typical values have been found to be around 25 ppm/°C, with a close-to-linear variation [33]. Devices developed by our research group have produced higher TCFs of >35 ppm/°C [35]. When used for thermal sensing, it is important to consider the heat losses through radiation, conduction and convection to the surrounding environment and through the contacts used to suspend and connect the device. The temperature change therefore, is affected by the connections and medium surrounding the resonator. For IR
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sensing applications such as presented here, the heat losses should be kept to a minimum in order to ultimately maximize the heat confined to the structure and in turn produce a larger temperature/frequency change. This may be done by minimizing the size of the anchor electrodes to increase their thermal resistance and reducing the air pressure around the resonator by encapsulating the device in a vacuum. In this thesis, a device (developed in [36]) that was not under vacuum encapsulation, but had higher thermal resistance in the anchor (thinner anchors) compared to previously developed devices in the group [37] was used.

3.1.1 Graphene-Aluminum Nitride Resonator

In this work, a resonator with graphene as the floating electrode (from [36]) was implemented as an IR sensor. Previous devices ([37]) developed in the group used a \SI{100}{\nano\meter} layer of gold as the floating electrode. If gold was the floating electrode for use as an IR sensor, it would be extremely ineffective because gold reflects IR almost completely. Graphene is used as the electrode because, not only is it more conductive than gold (almost 3x), it is also almost completely transparent, allowing the IR radiation to enter the structure itself. It is also possible that the graphene layer by itself absorbs some of the IR, as described in [38] due to surface plasmons. The thickness of the graphene layer is extremely small, and is ideally just one atom thick. It is only held to the AlN layer through van-der-waals forces and therefore does not mechanically load the resonator structure unlike a heavy gold layer, allowing for higher resonant frequencies and possibly better quality factors (sharpness of resonant peak) [37]. Thus the effectiveness of resonators as thermal sensors and the performance enhancement afforded by graphene proved instrumental in forming the idea to investigate these resonators as sensors for IR spectroscopy.

3.2 The Spectroscopic Measurement Setup

The aim of the work done using the resonator was to prove that the resonator, when sensitized to absorb IR, is able to determine, with a good level of certainty, differences in the IR signal power transmitted across a translucent sample (a liquid solution here) for varying concentration of the analyte in the sample. Thus, the measurement setup has three main parts: the source, the sample holder and the sensor. The source used was a \SI{5}{\mu m} wavelength Continuous Wave (CW), \SI{70}{\milli\watt} QCL (uncollimated) borrowed from EOS photonics, Massachusetts. The laser beam was invisible and therefore was located using thermal paper purchased from Thorlabs Inc. Since the beam
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was uncollimated and non-parallel to the optical table on which the system was set up, alignment and focusing was done to pass the beam exactly through the center of the sample and achieve maximum irradiation on the sensor. A visible, collimated red laser was coaxial to the IR laser to roughly estimate the invisible laser beam location. The first step in setting up the system was to align the laser to a particular height, and parallel to the optical table. This was done using the ‘U’ fold method, which consists of two beam steering mirrors (called kinematic mirrors) that are adjusted iteratively in order to pass the beam through two small apertures that define the optical axis. As shown in Figure 3.4, two of these alignment procedures are required: one for the IR laser and the other for the visible red laser. Two independent mirrors are required for each beam because both the angle, and the position of the beam needs to be set.

![Diagram](image)

Figure 3.4: Beam alignment and co-alignment setup

The aim of alignment is to send the beam through two parallel apertures whose height determines the height of the required beam. The aperture centers are kept at the same height to make the optical axis parallel to the optical table. Visible laser alignment was done by keeping a thermal sensor (Thorlabs S302C) at the “sensor” position in Figure 3.4 and at first, roughly aiming the beam to pass through the two variable apertures (iris) kept open at around 7.5 mm diameter. Then the beam is steered by separately adjusting M1 and M2 for the visible laser and M3 and M4 for the IR laser for smaller apertures while the power on the sensor is monitored. A number of iterations are required, because the steering done to move the beam position affects the angle as well and the position of the beam on the second mirror. The iterations are stopped and the beams are considered aligned when the power reading decreases for any adjustments made to the kinematic mirrors when using the smallest apertures. Co-alignment is made possible through the use of a dichroic mirror for
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the IR laser, which reflects IR but transmits visible light.

After the beams are co-aligned, the optical axis of the IR laser is made visible by the red laser. Now the sample and sensor can be positioned such that both are collinear to the optical axis. The samples considered for analysis here is a 1 mm path length liquid solution whose concentration is varied while measuring the change in the response of the sensor. The setup is done without affecting the alignment done previously as shown in Figure 3.5, and the aperture and thermal sensor are replaced by an optical chopper, sample cell and the G-AlN resonator. The aligned beam passes through an optical chopper to provide a base line for the received signal. It then passes through an FT-IR liquid cell (a 1 mm path length sealed liquid cell from International crystal) with ZnSe windows, which contains the analyte. A convex lens ZnSe is used to focus the beam to a minimal size to maximize the power received by the sensor, which is positioned in the optical axis where maximum response occurs. The final measurement setup is shown in Figure 3.6.

3.2.1 The Analyte and Sample Preparation

The aim of the measurement was to send an IR beam of constant power through a liquid solution (the analyte) and focus the beam on the G-AlN resonator and to see if there was a detectable change in its response for different concentrations of the solution. This first required the careful
Figure 3.6: Picture of the optical table setup for measurement
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selection of a solute and solvent such that the solvent was IR transparent, while the solute had finite absorption in the wavelength of interest (5 μm).

In the initial phase, it was planned to use water as a solvent and potassium ferrocyanide (which has strong absorption at 5 μm) as the solute. Unfortunately, it was found that when the 1 mm liquid cell was used, there complete absorption of the IR in the liquid cell itself and there was no radiation exiting the cell even for pure water, rendering the liquid cell unusable for water-based solution. But since the objective of the work done here was to test the spectroscopic application of a G-ALN IR sensor, the actual mixture did not matter as long as the solvent had very little absorption for the 1 mm path length and the solute was more IR absorbent than the solvent. Thus, based on practical measurement with different liquids available in the lab, it was found that acetone was the most transmissive compared to two other liquids: pure water and pure IPA. This was supplemented by FT-IR data from online databases such as Sigma-Aldrich.

Shown in Figure 3.7 is the transmittance spectra (in %) of the three pure liquids. Water is represented by the black curve, IPA by the brown and acetone by the blue. It can be seen that acetone has 95 % transmittance compared to the 80 % of water and 93 % of IPA. Thus, from these data and observations, it was decided that the solvent will be acetone, while the solutes will be pure water and pure IPA. It is surmised that the path length of 1 mm is too much for pure water as its optical depth (its absorption coefficient at 5 μm \(k_v\) times \(l\) (path length)) becomes large enough to reduce the power exiting the liquid cell to negligible values (see the Beer-Lambert’s law in equation 2.16). Since the path length could not be decreased, it was not possible to test water-based solutions. Thus, Acetone-based solutions were used because of its smaller absorption coefficient and optical depth for the same 1 mm path length. The solutions were prepared using 100 mL volumetric flasks and the concentrations were determined by numerical value of the solute added. First, the solute (water or IPA) was measured using graduated cylinders and was added to a half-filled volumetric flask containing acetone. The, the volumetric flask was filled up to the 100 mL mark with acetone to get a solution of concentration: \((100 - X)\) %, where \(X\) is the numerical value of the volume of solute added. Solutions with concentrations between 0.25 % to 20 % were prepared and sequentially tested, making sure that interference from previous samples was avoided.

3.2.2 Measurement Method

The prepared solutions were loaded into the liquid cell using two luer tip syringes: one with the sample solution connected to the inlet (see Figure 3.5) and the other connected to the outlet.
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IR Transmittance/Absorbance Spectra of Acetone, H$_2$O and IPA

Figure 3.7: Comparison of the relative transmittance of acetone, water and IPA (Source: Sigma-Aldrich)

The latter is used to pull the sample into the cell, since pushing it could damage the seals. After the cell is filled without any air bubbles, the syringes are removed and the measurement from the sensor is taken. Each measurement is followed by a wash of the cell using acetone and then emptying the cell after which the next sample is loaded.

A network analyzer is used to read the sensor output. It configured to continuously monitor the admittance of the resonator at a particular frequency. This frequency is the resonant frequency when there is no signal. Shown in Figure 3.8 is the admittance curve that was measured for the device tested (in blue). When there is a change in the temperature of the resonator, the resonant frequency changes. This is reflected in the admittance curve as shift of the curve to the side. Thus, to measure the frequency shift, first a point in the middle of the curve, where the slope is steep and more or less constant is selected (Here it was $f_0=1.308675738$ GHz) and the admittance is noted as say ‘$A_0$’. another arbitrary point on the slope is selected and the slope is calculated from the two data points as ‘$m$’. The network analyzer is then configured to monitor the admittance at the center frequency. As can be imagined, when the curve shifts to the left due to a corresponding temperature change (in brown), the admittance at the monitored center frequency drops to an admittance ‘$A_1$’. Thus, the frequency shift can then be found out by the relation:

$$\Delta f = f_1 - f_0 = m(A_1 - A_0)$$
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The final measurement is in parts per million (ppm), which is given by:

\[
\Delta f \text{(ppm)} = \frac{\Delta f}{f_0} \times 10^6
\]

The ppm shift can be plotted as a time domain signal with the help of an optical chopper which helps to set a base line for the sensor output when IR signal is OFF. The resonator is quite sensitive to the external environment (including the human body if it is too near) and tends to fluctuate the resonant frequency even when there is no IR input. This is the main reason why a chopped signal is required and the measurement is done as a frequency shift and not as an absolute frequency.

3.3 Results

The measurements from the network analyzer were converted into ppm shifts in frequency. The results for the water-in-acetone measurements is shown in Figure 3.9 as a time domain signal, corresponding to the optical chopping frequency. As seen, the ppm shift in frequency decreases with increasing concentration, with a maximum detectable concentration of 10% and a minimum detectable concentration of 0.5%.

In order to qualitatively establish the utility of the resonator as a spectroscopic sensor, measurements with IPA-acetone solutions were also done, because it has a smaller absorption...
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Figure 3.9: Measured response for water-in-acetone solutions

at 5 µm than water, as shown in Figure 3.7. Thus the expected response as compared to the water-acetone mixture was a much lesser shift in frequency across the same concentration range. The measurements done with the mixture as shown in Figure 3.10 agree well with the expectations, showing hardly any changes for smaller concentrations and becoming more noticeable only past 7.5% concentration. The ppm shift versus concentration at a particular instant in the ON region for both the water-acetone and IPA-acetone solutions is compared in Figure 3.11. It clearly shows, that the IPA system’s response, because of its lower absorption, varies less with increasing concentration, compared to that of the water-acetone mixtures. This matches well with the expected trend shown in Figure 2.15, thereby establishing that the sensor works well and predictably in a spectroscopic system such as the one presented.

3.3.1 Discussion

The experiments performed in the previous section have demonstrated an application of G-AlN resonant IR sensors as a sensor for IR spectroscopic studies. Due to limited equipment and time, measurements were limited to the wavelength provided by the source, which was a 5 µm QCL. Even with no characteristic absorption features for the two solutes in this region of the
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**Figure 3.10:** Measured response for IPA-in-acetone solutions

**Figure 3.11:** Comparison of measured responses for water-acetone and IPA-acetone for different concentrations
spectrum, it was possible to differentiate between two different chemicals that had only a small difference in absorbance at 5 µm. This is indicative of the potential of this sensor to be used in different wavelengths which are characteristic of a specific chemical, thereby making it a specific chemical sensor. Since the transmitted power is exponentially related to the product of the chemical’s absorptivity and path length, it is expected that if a chemical with strong absorptivity is dissolved in the same liquid cell setup, there will be much larger change in frequency shift for the same change in concentration compared to the water-acetone setup. This would imply that the sensor would be able to resolve smaller changes in concentration and also allow for a lower limit of detectable concentration.

Both the mixtures used for measuring the response have a lower and higher limit of detection. The lower limit of detectable concentration is determined by the strength of absorption at that wavelength. If the absorptivity of the solute is low, then its concentration should increase in order to be able to observe a deviation from the pure solvent case. On the contrary, if it has high absorptivity, a smaller concentration can provide the same deviation. It is also affected by the noise of the system as shown in Figure 3.9, where there is clearly visible noise. The higher limit of detectable concentration depends on the noise as well. It also depends on the path length of the medium and source intensity. The sensitivity of the device itself affects the overall range and resolution as well. The resonator used was not a perfect absorber at 5 µm, rather it was only around 20 % absorptive at 5 µm as inferred from its FT-IR reflectivity spectrum. Thus, if the source was a wavelength matching an absorption line of the solute, and the sensor was 100 % absorbing, it would be a much more effective spectroscopic sensor. In fact, such a device was planned to be used (metamaterial-enabled resonator) but was damaged before testing.

The optical components used in the beam path also affect the response due to reflection and absorption. The liquid cell, which has two windows of ZnSe have a large amount of reflectance due to its relatively high refractive index and multiple interfaces where reflection occurs. This, couples with around 85 % transmittance implies a signal power loss at the exit. The only way to counteract the effect is to coat the windows with and anti-reflective (AR) coating, which was an unavailable option. The focusing lens on the other hand, was an aspheric AR coated lens. Almost half the power was lost in the beam before it reached the sensor (from 71 mW to 36 mW), when pure acetone was used. Thus, the effect of optical components must not be ignored in future experiments.
Chapter 4

A Proposal for a Zero Power Exhaust Plume Detector

This part of the thesis proposes a novel sensing system, for vehicle detection based on the IR radiation emitted by the exhaust plume of vehicles. The aim is to develop a system that has zero power consumption, relying only on the IR radiation to be detected as the power source. It is expected to have approximately zero power consumption due to the detection mechanism used, incorporating the sensor, signal processing and output generation in one single structure based off a thermomechanical bimaterial switch.

4.1 Need for A Zero Power Detector

The proposed design is aimed at detecting hot gases at a distance based on the emission spectrum of the gases using a thermomechanical switch that closes upon absorbing IR radiation from the gas. Designed to be a completely passive system, (meaning not requiring a background source or a power supply for detection), it is a novel, minimalistic approach to long range gas detection that currently use complex detection systems and signal processing. Currently, there are only expensive techniques for detecting gas plumes at a distance without using a background source illumination such as blackbodies of lasers. But almost all these methods are characterized by high complexity in data processing and cost and are not typically used in a multi-node system. Wide area measurement is done by a FT-IR imager-scanner mounted on a tripod or aircraft [39]. These are versatile instruments capable of extreme sensitivity, high signal-to-noise ratio, precision and long range measurements (kms) finding use in the military and environmental monitoring agencies.
for a variety of purposes including national security and weather prediction. It is an extremely powerful tool when used for hyperspectral imaging over a wide area as it is not only capable of identifying a gaseous species, but also provides a visual spatial distribution of the gas of interest as an image overlay, highlighting the regions where the gas is present. Since it has 3D data (x, y, spectrum), multiple species of gases can be imaged by selecting the spatial data corresponding to the characteristic spectrum of the gas.

Since the application objective for this sensor is not in measurement but in gas detection, it does not require the cumbersome paraphernalia of other techniques. It does not require a source and incorporates the wavelength filter functionality of conventional spectrometers as a plasmonic narrow-band absorber layer on the surface of the sensor itself, greatly reducing system complexity. Thus, it occupies a unique spot in applications requiring passive detection where battery power is a precious resource. For example it may be deployed at a number of spots in a remote location and used as an activation system for an alarm, RF communicator or other device that is powered, but does not need to be ON unless something is detected. The footprint of the device is also of concern to such remote, multi-node applications, since if it needs to do the detecting, it is best if detector itself does not get detected in the process. To the best of knowledge, there are no such zero power gas detectors till date.

Thus the proposal for such detection system is extremely appealing and has motivated the interest toward realizing the system practically. The work presented here documents the concept and theoretical models used to predict its limitations, feasibility and possible improvements. It also includes a proposal for testing the system after fabrication.

4.2 Concept: Detection of the IR Spectroscopic Signature of Gases

As mentioned in Section 2.3.3, in case of purely emissive systems, the radiance or emission spectra depend on the the temperature of the object radiating and its wavelength-dependent emissivity. Thus, a hot gas such as CO$_2$ emits radiation only in certain narrow regions of the IR spectrum. This selective emission can thus be manipulated to work as say, a CO$_2$ sensor.

Engines using gasoline or diesel as fuels typically produce the following compounds on combustion: CO$_2$, CO, NO$_x$, H$_2$O, SO$_x$ and Hydrocarbons such as Methane [40]. Natural phenomena, even fires typically do not produce these gases simultaneously in a localized position. Thus, the emission spectra of the heated gases coming out of the tailpipe of a vehicle can by itself act as a signature specific to a fuel-burning vehicle. In the infrared region of the electromagnetic spectrum,
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all the above gases have well-defined emission regions that may be exploited to detect an exhaust plume’s signature. A simulation of the emission spectra of the constituents in a gas plume has been shown in Figure 4.1.

![Emission Spectra of Exhaust gases](image)

Figure 4.1: The simulated emission spectra of vehicle exhaust, showing clear spectroscopic signature wavelengths of constituent gases

### 4.2.1 Detection Technique

The idea here is to make multiple spectrally selective detectors, each corresponding to a certain emission feature of a gas (these detectors will be referred to as in-band detectors). The detectors are connected in a logical circuit that would provide a logical 1 only when these gases are detected. Care is also taken to avoid detection of background radiation and other emitting objects, since they may also emit in the same spectral regions as a target gas. The difference is that since they also emit at non-signature wavelengths, by allocating detectors for these out-of-band regions, a logic can be formed as follows:

\[
\text{if}((\text{inbanddetectors} == 1)\&\&(\text{outofbanddetectors} == 0))
\]

\[
\text{output} = 1;
\]

\[
\text{else}
\]

\[
\text{output} = 0;
\]

\[
\text{endif}
\]
CHAPTER 4. A PROPOSAL FOR A ZERO POWER EXHAUST PLUME DETECTOR

The in-band detectors are designed as normally open (NO) switches and the out-of-band detectors will be designed as normally closed (NC) switches. Connecting them in series as shown in Figure 4.2 would then allow them to perform the function of selective detection of gases and avoid false detections. Each switch representing a gas in figure consists of multiple NO switches in series, each sensitive to one emission feature of the gas. Thus, since the circuit uses physical switches and not devices like transistors, there is no channel for leakage currents to occur to leak power from the logic voltage at one end provided by a generic lithium battery. The switch structure is described in the following section.

4.3 Detector Design

A variety of thermomechanical switches in literature were surveyed in Section 2.4.3, from which it was decided that the detector design will be based on an uncooled, temperature and stress-compensated structure as developed used in [20, 41]. The design uses two pairs of bimetallic arms, the inner pair for detecting IR, and the outer pair for compensating for ambient temperature.

As shown in Figure 4.3, it consists of a detecting head for absorbing radiation, an inner pair of bimetallic beams which, because they are attached to the absorbing head, bend when the detector head heats up. These are anchored to the substrate using two identical outer bimetallic beams that are thermally isolated from the beams that bend due to absorbed IR. Thus, the signal from the incident IR is confined within the inner pair of beams, effectively decoupling the detector head from ambient temperature effects. When there is a change in ambient temperature without any IR input, both the inner pair and outer pair bend and maintain the z-position (axis perpendicular to the figure) of the edge of the detector (marked ‘X’). Then when a signal reaches the detector, the detector transmits the heat to the inner pair of legs, increasing its temperature and causing a further bending of only the inner region. This causes the point marked ‘X’ to move in the z-axis. As a visual example, an example of its working, from [41] is shown in Figure 4.4 where the main structure is
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Figure 4.3: Schematic of the top-view of the bimaterial sensor used in [20]

shown in Figure 4.4 A, the effect of ambient temperature in Figure 4.4 B and effect of IR radiation falling on the head in Figure 4.4 C.

When this structure is used as a temperature-compensated IR sensor, ideally, the edge of the detector head marked ‘X’ should have no displacement of ambient temperature fluctuations. But in practice, the device developed in [41] suffers from small displacements of up to 200 nm for ambient temperature changes of 20 K. In order to compensate for this non-ideality, it was decided that there will be two almost identical, independently moving structures facing each other. Therefore, if it has the same sensitivity as the above device, both of the detector heads will move 200 nm for an ambient 20 K change, and the relative movement, which is the parameter of interest, will be very close zero. If one of these heads are made spectrally sensitive, and if the right IR wavelength is incident on the detectors, the detector with the sensitized layer will absorb more IR and thus bend towards the non-IR absorbing detectors. If the power of the IR is high enough, the bending will be large enough to make the two detectors come in contact. With appropriately positioned contacts on the detector edge, these two detectors can act as a mechanical normally open switch. This proposed structure is represented schematically in Figure 4.5. ‘Ti’ is the thermal isolation region and ‘Tin’ is a thermal isolation region with a thin metallic layer to provide electrical connection to the switch region in the middle of the structure.

As shown in the inset of Figure 4.5, the type of switch that will be used for the in-band
CHAPTER 4. A PROPOSAL FOR A ZERO POWER EXHAUST PLUME DETECTOR

Figure 4.4: SEM image of the working of the bimaterial cantilever when there is no thermal stress (A), when there is thermal stress due to ambient temperature (B) and when there is thermal stress due to received IR signal (C) (Source: [41])

Figure 4.5: Schematic of the proposed detector-switch structure. Inset shows the switch structure
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detectors is a NO type switch where P and R form the two terminals of the switch and Q is used to close the switch. As shown, detector D2 has a metamaterial IR absorbing layer that selectively absorbs the IR. Thus, with increasing ambient temperature, the separation of the contact, PR and Q, remains constant. When IR is absorbed by D2, the position of PR remains the same, but Q, which is attached to D2, moves down. With the right amount of power, Q connects P and R and closes the switch.

For a NC switch that will be used for out-of-band detection, the same structure will be used, but the contacts will be fabricated such that they are in contact normally. Thus, if the switch is to open the circuit upon reception of out-of-band IR wavelengths, the detector D1 should bend down upon on receiving IR radiation. Thus, the IR sensitive layer must be on D1 for the structure to act as a NC switch.

4.4 Received Power Estimation

In order to design the structure of the switch with the right sensitivity, the first data that is needed is the expected power reaching the detector head for different distances. On order to find that, the power emitted by the hot gas in the plume to which detector will be sensitive to, must first be known. This was estimated using the concepts outlined in Section 2.3.3 for the emission case. Here an example of CO\(_2\) is taken as the species to be detected. From the emission spectrum of CO\(_2\) shown in Figure 2.16, there are multiple emission features in the radiance spectra. For the represented example, the emission feature at the 4.26 µm region (2350 cm\(^{-1}\)) is considered and the subsequent calculations are based on this. In order to find the power reaching the detector, first the radiance/ emission spectra was calculated for a CO\(_2\) for a set of parameters such as temperature, pressure and its partial pressure using the equations described in Section 2.3.3.

The following assumptions were made for estimating the radiance of the CO\(_2\) plume at its surface: the total pressure of the exhaust plume is 1 atm; the partial pressure of CO\(_2\) is 0.1 atm; the plume is modeled as a cylinder of height and radius 8.1 cm (representing the section of the exhaust plume next to the tailpipe where the temperature is highest) with its axis normal to the detector and the temperature is between 600 K and 800 K, which corresponds to a probable range for vehicles like pick-up trucks that are equipped with diesel particulate filters.

Once the radiance/ emission spectra was calculated, it was treated as a source illumination for an absorptive system that fills the intervening space between the plume and the sensor. The radiance as seen by the detector would be affected by the intervening medium. Thus, using Beer-
CHAPTER 4. A PROPOSAL FOR A ZERO POWER EXHAUST PLUME DETECTOR

Lambert’s law (equation 2.16 the actual apparent radiance is calculated. The assumptions made are: the intervening distance contains no gaseous species other than CO₂, that absorbs considerably in the wavenumber range of interest (around 2350 cm⁻¹); the pressure of the medium is 1 atm; partial pressure of CO₂ which is the sole absorber is 0.00039 atm; there are no particles to introduce scattering effects and the temperature is 296 K. The simulated radiance at the plume and as seen by the detector is shown in Figure 4.6. As is evident, the absorption of intervening atmospheric CO₂

![Image of Figure 4.6: Plot of apparent radiance seen by detector compared to the radiance seen at plume surface]

is quite substantial. This spectral radiance is then integrated over the wavenumbers comprising the emission feature using:

\[ L_{\text{eff}} = \int_{\nu_1}^{\nu_2} L_a(\nu, T) \, d\nu \quad (4.6) \]

where the \( L_{\text{eff}} \) is the integrated apparent/effective radiance seen by the detector, \( \nu_1, \nu_2 \) is the range of integration (2200 cm⁻¹ to 2400 cm⁻¹ here) and \( L_a(\nu, T) \) is the apparent radiance at wavenumber \( \nu \). The integrated radiance (unit: W/cm² sr) gives the power emitted per unit area per solid angle. The power received by a collecting optic such as a convex lens or a concave mirror kept at a distance \( r \) from the plume is then calculated as:

\[ P_l = \frac{L_{\text{eff}} \times A_l \times A_p}{r^2} \quad (4.7) \]
where, $P_l$ is the power received over the area of the collecting optic, $A_l$ is the area of the collecting optic, and $A_p$ is the area of the plume surface visible to the collecting optic. The solid angle is given by: $A_p/\pi^2$ (unit: steradian [sr]). The next step is to calculate the actual power received by the detector. Since the plume is an extended source, the image formed by the optic (at the point in the optical axis with the highest power density smallest image size) has a finite size determined by basic Gaussian lens/mirror laws.

$$\frac{1}{f} = \frac{1}{d_i} + \frac{1}{d_o} \quad \text{(4.8)}$$

$$\frac{d_i}{d_o} = \frac{y_i}{y_o} \quad \text{(4.9)}$$

where, $d_i$ is image distance, $d_o$ is object distance, $f$ is focal length, $y_i$ is image size, $y_o$ is size of object. Thus, the image size decreases as the plume’s distance increases approaching the smallest size when the plume is at infinity. Since at shorter distances the image size may be larger than the detector (which is a square shape of side 200 $\mu$m), the actual power received at these short distances is only a fraction of the power distributed over the image area. This is given by:

$$P_d = P_l \times \frac{A_d}{A_i} \quad \text{(4.10)}$$

where, $P_d$ is the power falling on the detector, $P_l$ is the power collected by the lens into the formed image of area $A_i$ and $A_d$ is the area of the detector head. Both a lens based system and a concave mirror based system were considered and the expected power received by the detector for a distance of up to 160 m was estimated using all the above equations.

4.4.1 Results for Estimated Power at detector and Discussion

The simulation of expected power reaching the detectors were done for two different plume temperatures for both a lens-based system and a mirror-based system. The temperatures were 600 K and 800 K and the diameters of the optics used were 2.5 cm (2.5 cm focal length) for the convex lens and 15 cm (5.625 cm focal length) for the concave mirror. Both these results may be relevant to different applications which may need a long range detector but a small footprint, or a long range detector where the system footprint is not critical. Figure 4.7 shows the final power expected at the detector head. Note the discontinuity in all the cases. This corresponds to the plume distance when the image are becomes equal to detector area. After this point, equation 4.10 is not valid anymore, and the curve follows equation 4.7.
For a possible detector size of 200x200 µm² and an estimated detection threshold of 500 nW, it is seen that, the lower limit of range of detection for a plume of 600 K corresponds to the lens case as expected. It is expected that this system would be able to detect a CO₂ plume at 600 K at a maximum of 5 m. The upper limit for the detection system corresponds to a plume of temperature 800 K with a 15 cm diameter mirror. It is expected that if these were the conditions, it would be able to detect a CO₂ plume at 60 m. Simulations up to 160 m is done to provide a horizon for development to work towards: It is aimed that the device would be made sensitive down to 50 nW IR radiation in the future, which would correspond to a 140 m distance. It is interesting to note the effect of a larger optic when compared to the the 2.5 cm lens case for a given plume temperature and distance. For example, at 19 m, where the lens case receives 50 nW, the mirror system provides a power of 1.74 µW, which is almost a 35x increase, allowing for a much farther range of detection of 78 m with the mirror. Thus both of the optics afford potential feasibility for different detection distances that fall well within certain application requirements such as for vehicle detection, explosion detection and aircraft detection.
CHAPTER 4.  A PROPOSAL FOR A ZERO POWER EXHAUST PLUME DETECTOR

4.5 Proposed Testing Technique

Once the devices are fabricated, they will be tested in the lab by irradiating the detectors with a source that mimics real exhaust plumes. First, the spectral response and hence the spectral selectivity of each detector will be characterized using a FT-IR microscope. After devices are ensured to have the required characteristic, a calibrated blackbody source with a controllable temperature range between 320 K and 1320 K will act as a broadband source that passes IR radiation through a narrowband transmission filter placed at the aperture of the blackbody, resulting in an output radiance that mimics a particular gas species, based on the center frequency of the filter. Each detector can be tested this way sequentially for different distances by using a filter wheel with multiple passband ranges corresponding to specific gases and out-of-band emissions. The test setup schematic is shown in Figure 4.8.

![Figure 4.8: Schematic showing proposed testing plan using a blackbody and filters. The filters will moved in front of the blackbody aperture sequentially](image)

The sensing system will also be tested by using a heated gas cell (GS05670 available from Specac) whose composition, temperature and pressure can be controlled. All currently available commercial gas cells can only be heated up to a temperature of 520 K. Thus, this setup will be used for testing the overall response of the system at short distances, complementing the blackbody test setup, which has a higher temperature but can only mimic one emission feature and hence test only one detector at a time. The results for the lower temperature source can be approximately scaled to a higher temperature cell mathematically as well. The test setup used is schematically shown in Figure 4.9. The distance between the detector and gas cell will be varied and the whole system will
be monitored for a response from the logical detector circuit. To mimic non-exhaust emissions, hot objects or other gases will be used, which would allow to characterize the system’s accuracy.
Chapter 5

Conclusion

Two spectroscopic systems are presented in this thesis. The first is a proof-of-concept type treatment of testing a G-AlN resonator as an IR sensor for absorption-based spectroscopic analysis. The other one is a proposal for developing a novel method of spectroscopic detection of vehicle exhaust plumes while consuming zero power. The latter uses the concept of emission spectroscopy, relying on the energy of the signal alone for detection. Thus, both these works complement each other in many ways. These are noted in Table 5.1. From the positive results obtained for both systems, further research is planned to develop them to attain commercial application.

<table>
<thead>
<tr>
<th>The Zero Power System</th>
<th>The G-AlN Sensor System</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unpowered</td>
<td>Powered</td>
</tr>
<tr>
<td>Passive – No requirement of background source</td>
<td>Active – requires a background source</td>
</tr>
<tr>
<td>In-built signal processing and temperature compensation</td>
<td>External signal processing and temperature compensation</td>
</tr>
<tr>
<td>Emission based</td>
<td>Absorption based</td>
</tr>
<tr>
<td>Only does detection</td>
<td>Can do both detection and measurements</td>
</tr>
<tr>
<td>Detection limited by distance of object</td>
<td>Detection limited by type of source: Lasers can provide for much greater distances</td>
</tr>
<tr>
<td>Designed for detection of a narrow range of chemicals</td>
<td>Can be used for analysis of any chemical</td>
</tr>
<tr>
<td>Relies heavily on spectral selectivity</td>
<td>Spectral selectivity can be done externally and can used for broadband analysis</td>
</tr>
<tr>
<td>Minimal complexity and external components</td>
<td>Requires external components for power and data conversion</td>
</tr>
</tbody>
</table>

Table 5.1: Comparison of investigated systems
CHAPTER 5. CONCLUSION

5.1 Work Summary and Future Work

5.1.1 The G-AlN Absorption Spectroscope

A G-AlN resonator developed recently by our research group [36], noted for its weak absorption of IR was investigated as a potential sensor for incorporation in spectroscopic system. To test the sensor, a measurement system based on NDIR-type liquid absorption spectroscopy was designed. The reference liquid was acetone which was found to have the highest transmittance at 5 µm, the source’s (QCL) wavelength. The response of the sensor, measured as ppm shifts in resonant frequency, was measured for solutions with varying concentrations of H₂O or IPA in acetone. It was found that the sensor was successfully able to differentiate between two chemical species - H₂O and IPA- with different absorptivities based on their response to IR radiation of constant power and wavelength. It was also observed that the sensor behaved in an expected manner, showing a larger variation for increasing concentrations of H₂O, which has a higher absorptivity, compared to the same increases in concentration of IPA in acetone. This proved the concept of using IR-sensitive G-AlN resonators as spectroscopic sensors.

The device used was not a strong absorber of 5 µm IR radiation, but still worked well in being able to sense the difference between varying concentrations of chemical species and different chemicals. It is expected that if the sensor was more absorptive at the wavelength of interest, it would be able to provide a higher response to the same intensity of IR, implying higher resolution measurements with better noise performance and concentration detection limits. It is planned to further investigate these resonant IR sensors for better spectral sensitivity by incorporating better-absorbing layers such as metamaterials or perfectly absorbing Fabry-Perot optical cavities which would imply improved noise performance and reduce external components. The measurement setup designed here is a first for our research group and its encouraging results as a spectroscopic system establishes a firm foundation for further work. Plans for future work include development of higher sensitivity resonators with better thermal energy retainment, encapsulation of such devices in vacuum to reduce heat loss through convection and development of broadband IR sensitive resonators. The work done with resonators has opened two paths of IR sensing: One going towards development of IR spectroscopic sensors ending at a novel hyperspectral imaging system, and the other towards developing thermal sensors ending at a high speed, high resolution IR imager.
CHAPTER 5. CONCLUSION

5.1.2 A Zero Power Exhaust Detector

A novel, zero power vehicle exhaust plume detector technique was proposed in this part of the thesis. The system is based on the property of gases having a specific emission signature characteristic to its molecular structure. Thus, by using an array of detectors sensitive to exactly one of these signatures, and connecting them in such a way to give an output only in the presence of a specific combination of gases, it can be made to respond, with a high degree of certainty, to only the required mixture, such as an exhaust plume. The device working mechanism and its ability to almost completely compensate for ambient temperatures has also been explained. Since the exact design and working distance requires the estimation of the power received at various distances, calculations based on reasonable assumptions was done, incorporating as much accuracy as possible, like considering atmospheric absorption effects and how the size of the detector and the size of the optics used affect the final power received by the detector. Estimation of power at detector was done for two systems using the same detector: one with a 2.5 cm diameter mirror, and the other with a 15 cm diameter mirror. It was found that for a possible device size of 200x200 µm² with detection threshold of 500 nW, the maximum range of detection is about 60 m, for a plume temperature of 800 K and collecting optic size of 15 cm. It is expected to extend this range to 140 m for a detection threshold of 50 nW. Even higher ranges are predicted for hotter plumes like from jet engines.

The positive results obtained from the theoretical analysis done here has established a firm background for the succeeding stages of progress towards an actual working model. There is no such device available currently that uses such a mechanism for detection while also consuming zero power. The completion of a working model could possibly be a game-changer for military sensing or environmental monitoring applications that need to preserve battery power until absolutely needed. In fact, a possible application could be the use of both systems presented in this thesis working in tandem. For example, if vehicles passing along in a highway need to be monitored for some exhaust gas pollutants, the zero power detector would first detect if the pollutant species are present; On detection it will turn on the secondary measurement system which is an IR source and the demonstrated IR resonant sensor positioned on opposites sides of the road, as shown in Figure 5.1. The source passes IR radiation through the exhaust plume of the passing vehicle and calculates the concentration of pollutants based on the transmission spectrum. Thus, the whole system can have low average power consumption, needing it only when the pollutants are detected by the zero-power sensor, which switches it on.

Though it has been specified here to be used as a vehicle plume detector, it is equally
possible to use it for other applications like explosion, gunfire and forest fire detection. It could also potentially be used as a one-bit color depth passive hyperspectral imager. It would use a focal plane array of detectors sensitive to only one hot gas species. When a plume appears in its field of view, it would provide a two-color image of the plume with the sensors exposed to the IR being closed while the others in the array staying open. Future work would focus on first fabricating a preliminary device to observe any unpredictabilities in the final structure functioning and issues with fabrication. It is already expected that stiction forces could be a potential problem after the switch closes, not allowing the contact to return to the open position when there is no radiation. Other device-determined sensitivity enhancements will be investigated, one of which is to have a longer contact (Q- the middle contact), so that a small angular displacement at the edge of the detector will be translated to a large displacement at the end of the contact tip. Other methods could include developing more sensitive bimaterial combinations and bimaterial beam structures.
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