

Linear Cavity Tapered Fiber Sensor Using Phase Shift Cavity Ring Down Spectroscopy

by

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Down Spectroscopy**

Koç University

Graduate School of Sciences and Engineering

This is to certify that I have examined this copy of a doctoral dissertation by

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To My Beloved Family, Especially My Dear Mother

ABSTRACT

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This dissertation presents the linear optical cavity sensor based on fiber Bragg gratings and tapered optical fiber. The sensing and detection approach used in this thesis is phase shift cavity ring down spectroscopy. It is because of linear nature of the cavity and detection mechanism applied, that the whole study is named as “linear cavity tapered fiber sensor using phase shift cavity ring down spectroscopy”. First part of the thesis is dedicated to the study of experimental investigations that are available in the literature for the purpose of optical sensing. Among the reviewed techniques are the surface plasmon resonance (SPR), resonant wavelength shift, direct absorption spectroscopy, cavity ring down spectroscopy (CRDS) and its variants like cavity enhanced absorption spectroscopy (CEAS). Then these techniques are sorted out depending upon the advantages that they have on each other and limitations that put constraint on their performance. For example, surface plasmon resonance is a single pass approach, which is its major constraint. But multi-pass method dubbed as resonant wavelength shift, although it is sensitive enough but suffers from laser intensity changes. On the other hand, cavity ring down spectroscopy, is free from such errors but it is expensive and complicated technique.

The sensing technique called phase shift cavity ring down spectroscopy not only combines the advantages of CRDS and resonant wavelength shift but also provide solutions to their short comings. That is, it is economically feasible and not affected by the noise related to laser intensity. In the manuscript under study, mathematical foundation of the phase shift cavity ring down spectroscopy is also given due attention. In addition to this, fabrication of fiber Bragg gratings, their characterization, tapered optical fiber and its evanescent field are the topics which are also discussed in the same thesis. In the second part of the thesis, an experimental inves-

tigation has been reported by using tapered fiber based cavity. Here we employed a tapered fiber-based linear fiber cavity for the demonstration of a highly sensitive sensor for sucrose concentration in water using PS-CRDS. This, linear fiber cavity has a small cavity length (~ 1.25 m) that enables the observation and tracking of individual cavity modes in transmission and phase spectra recorded during laser sweeps. Hence, it eliminates the need for Pound–Drever–Hall locking of the laser source to the cavity resonance and thus provide a simpler experimental scheme. The analysis of the recorded data sets was performed to track the changes in phase shifts observed only when the laser wavelength is in resonance with the cavity modes. Such a mode-tracking PS-CRDS approach reveals limit of detection values less than around $400\text{ }\mu\text{M}$, better than the performance of previously demonstrated PS-CRDS sucrose concentration sensors employing fiber loop resonators. The sensitivity of our sensor critically depends on the fiber taper diameter and can reach up to around $6^\circ/\text{1mM Suc.}$ for $3.2\text{ }\mu\text{m}$ taper diameter at 6 MHz modulation frequency using fiber Bragg gratings (FBGs) with reflectivities around 86%. This value can be further increased by employing FBGs with higher reflectivities or fiber tapers with smaller diameters provided that the cavity loss due to water absorption is compensated with an amplifier. It is well known fact common sources of optical losses are scattering, optical components, splicing or water absorption (for liquid phase measurements). These losses become more prominent at small taper waist diameters. So, by compensating these losses, working at the taper diameter lesser than $2\text{ }\mu\text{m}$ is possible. Last section of the thesis explains the working of the system for above said idea. Basically, to compensate for the optical losses, a booster optical amplifier (BOA) was added in to the previously designed simple PS-CRDS experimental scheme. After carefully adjusting the gain, this optical amplifier, provides enhanced ring down time and more round trips of light into the cavity. Which ultimately manifests itself in the form of improved sensitivity and detection limit.

Some exemplary measurements performed with the amplified PS-CRDS showed the sensitivity of $4.84^\circ/\text{1mM Suc.}$ for $2\text{ }\mu\text{m}$ taper diameter at 2 MHz modulation frequency. Higher sensitivity can also be achieved by adjusting booster optical amplifier at higher gains and using even more thinner tapers. The thesis ends with the concluding remarks and future directions. It highlights the potential applications of PS-CRDS and suitable candidates to replace tapered optical fiber as a sensing head. Implementation of these changes further enhance the performance of sensing device.

ÖZETÇE

Evre Kaydırmalı Çınlaç Boşalım İzgegözlem Yöntemi ve Sündürülmüş Optik Lif Temelli Doğrusal Çınlaç Algılayıcısı

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Bu tez Bragg ızgaralar ve sündürülmüş life dayanan doğrusal optik çınlaç algılayıcısını tanıtmaktadır. Bu tezde algılama yöntemi olarak evre kaydırmalı çınlaç boşalım izgegözlem yöntemi kullanılmıştır. Doğrusal yapısı ve uygulanan algılama mekanizması sebebiyle tüm çalışma “evre kaydırmalı çınlaç boşalım izgegözlem yöntemi ve sündürülmüş optik lif temelli doğrusal çınlaç algılayıcısı” olarak adlandırılmıştır. Tezin ilk kısmı, literatürde mevcut olan optik algılamaya dayalı deneysel araştırma çalışmalarına dayanmaktadır. Gözden geçirilen teknikler arasında yüzey plazmon çınlaması (SPR), çınlaç dalga boyu kayması, doğrudan emilim izgegözlemi, çınlaç boşalım izgegözlemi (CRDS) ve çınlaç ile arttırılmış soğurma izgegözlemi (CEAS) bulunmaktadır. Bu teknikler, her birinin sahip olduğu faydalar ve performanslarını kısıtlayan diğer sınırlamalara bağlı olarak değerlendirilmiştir. Örneğin, yüzey plazmon çınlaması tek geçiş yaklaşımına dayanır, ancak bu özelliği en büyük kısıtlamasıdır. Çoklu geçişi esas alan çınlama dalga boyu kayması yöntemi ise yeterince hassas olmasına rağmen, lazerin şiddet değişiminden olumsuz etkilenebilir. Öte yandan, çınlaç boşalım izgegözleminde bu tür kısıtlamalar yoktur ancak oldukça maliyetli ve karmaşık bir tekniktir. Fakat evre kaymalı çınlaç boşalım izgegözlemi (PS-CRDS), CRDS ve çınlaç dalga boyunun avantajlarını birleştirmekle beraber diğer yöntemlerde karşılaşılan sorunlara çözüm sunmaktadır.

Bu yöntem, ekonomik açıdan uygulanabilir ve ayrıca lazer şiddetindeki salınımlardan olumsuz etkilanmemektedir. Bu tezde CRDS yönteminin matematiksel temeli de ayrıca ele alınmıştır. Bunlara ek olarak bu bölümde lif Bragg ızgara üretimi, incelemesi, sündürülmüş lif ve sönen alan incelenmesi yer almaktadır. Tezin ikinci bölümünde ise sündürülmüş optik lif çınlacı ile gerçekleştirilen deneysel çalışmalar yer almaktadır. Yaptığımız çalışmada PS-CRDS yöntemiyle sudaki sukroz derişimini

yüksek hassasiyette ölçebilen sündürülmüş lif temelli doğrusal çınlaç kullanılmıştır. Lazer taraması sırasında iletim ve evre izgözleminde elde edilen çınlama kiplerinin tek tek gözlemlenmesi ve takip edilmesi için çınlaç uzunluğu yeterince küçük tasarlanmıştır (~ 1.25 m). Bu sayede lazer kaynağının Pound-Drever-Hall kilitlenmesi ihtiyacı ortadan kalkmakta ve daha basit bir deneysel düzenek elde edilmektedir. Kaydedilen verinin analizi için lazer dalgaboyu çınlaç kipleri ile çınlamadayken evre kaymalarındaki değişimler gözlenmiştir.

Bu kip-takip PS-CRDS yöntemi ile yaklaşık $400 \mu\text{M}$ 'den daha düşük algılama hassasiyeti elde edilmiştir. Bu değer sakaroz derişimi algılaması için daha önce önerilen lif döngü çınlacı kullanan PS-CRDS performansından daha iyidir. Geliştirdiğimiz lif algılayıcının hassasiyeti temel olarak sündürülmüş lifin çapına bağlıdır ve yaklaşık $3.2 \mu\text{m}$ lif çapı için $6^\circ/1\text{mM}$ Sakaroz değeri elde edilmiştir. Burada lif Bragg ızgaralarının (FBG) yansıtma oranları %86'dır ve modülasyon sıklığı 6 MHz olarak ayarlanmıştır. Ölçülen sakaroz hassasiyeti FBG'lerin yansıtma oranlarının arttırılması ya da sündürülmüş lif çapının küçültülmesi ile daha da iyi değerlere çıkarılabilir. Sıvı ortamda ince lif çapı, suyun soğurmasından kaynaklı çınlaç kayıplarını arttırılabilir ancak bu kayıplar bir yükseltici yardımı ile baskılanabilir. Bu tip çınlaçlarda görülen kayıpların, saçılma, optik bileşenler, lif kaynaştırma ya da suyun soğurmasından kaynaklandığı bilinmektedir. Bu kayıplar daha düşük lif çaplarında daha da önemli hale gelmektedir. Bu tür kayıpları gidererek, $2 \mu\text{m}$ 'den daha düşük çaplarda çalışabilmek mümkündür.

Tezin son bölümünde, yukarıda bahsedilen şartların uygulanması ve sistemin çalışması örnek deneylerle anlatılmıştır. Temel olarak, optik kayıpları baskılamak amacıyla tasarlanan PS-CRDS deney düzenegine optik yükseltici (BOA) eklenmiştir. BOA, kazanç değeri dikkatlice ayarlandıktan sonra çınlaç boşalım süresini arttırmakta ve ışığın çınlaç içerisinde daha fazla dolaşabilmesini sağlamaktadır. Bu da ölçüm hassasiyetinin artmasına ve algılama limitinin daha da düşük değerlere ulaşmasına imkan sağlamaktadır. Yükseltici eklenmiş PS-CRDS ölçümlerinde $2 \mu\text{m}$ sündürülmüş lif çapı ve 2 MHz kipleme sıklığında $4.84^\circ/1\text{mM}$ sakaroz hassasiyeti elde edilmiştir. BOA kazanç değeri daha yüksek seviyelere çıkarılarak ve daha da ince sündürülmüş lif kullanılarak daha iyi hassasiyet değerleri elde edilebilir. Tezin son kısmında, elde edilen sonuçlar değerlendirilmiş ve gelecekte PS-CRDS'e dayalı algılayıcı ve algılama tekniğinin performansını arttırmaya yönelik önerilere yer verilmiştir. Bu kapsamda, PS-CRDS tekniğinin potansiyel uygulamaları ve ölçüm ucu olarak sündürülmüş lifin

yerini alabilecek uygun adaylar deęerlendirilmiřtir. Bu uygulamalarla geliřtirilen cihazın performansının arttırılması mmkndr.



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ABBREVIATIONS

APS-CRDS	Amplified Phase Shift Cavity Ring Down Spectroscopy
BOA	Booster Optical Amplifier
BW	Band Width
CW	Continuous Wave
CRDS	Cavity Ring Down Spectroscopy
DI Water	Deionized Water
EVF	Evanescent Field
EDFA	Erbium Doped Fiber Amplifier
EMI	Electromagnetic Interference
FWHM	Full Width Half Maximum
FSR	Free Spectral Range
FBG	Fiber Bragg Grating
FFT	Fast Fourier Transform
FP	Fabry–Pérot
FBG	Fiber Bragg Grating
HF	Hydrofluoric Acid
IPA	Isopropyl Alcohol
LOD	Limit of Detection
MMF	Multi Mode Fiber
OSA	Optical Spectrum Analyser
PCB	Printed Circuit Board
PS-CRDS	Phase Shift Cavity Ring Down Spectroscopy
PDH	Pound-Drever-Hall
RI	Refractive Index

RIU	Refractive Index Unit
SPR	Surface Plasmon Resonance
SMF	Single Mode Fiber
TIR	Total Internal Reflection
T-SMF	Tapered Single Mode Fiber
WM-DAS	Wavelength Modulation Direct Absorption Spectroscopy
VOA	Variable Optical Attenuator
σ	Absorption Cross-Section
d_p	Depth of Penetration Frequency
n_{eff}	Effective Refractive Index of Material
$\delta\lambda$	Full Width Half Max for Wavelength
n_h	High Refractive Index of Material
P_{in}	Input Power
I_{in}	Input Intensity
n_l	Low Refractive Index of Material
f_m	Modulation Frequency
α	Modulation Depth of Light Before Entering the Cavity
R	Mirror Reflectivity
N	Molecules per Unit Volume
α'	Modulation Depth of Light Exiting the Cavity
P_{out}	Output Power
I_{out}	Output Intensity
ϕ_{pp}	Peak to Peak Phase Shift
ϕ_{peak}	Peak Phase
T_{peak}	Peak Transmission
Q-factor	Quality Factor
t_r	Round Trip Time
τ	Ring Down Time Without Sample

τ_r	Ring Down Time With Sample
λ_r	Resonant Wavelength
D_{taper}	Taper Waist Diameter
L_{total}	Total Length of Cavity



Chapter 1

INTRODUCTION

Detection and sensing of change in any of the parameters, associated with the external environment or in the environment itself is of great importance. Such information can be used in lots of real world applications in the fields of chemistry, biomedical engineering, food and beverages, meteorology [Sun et al., 2003, Hahn-Hägerdal et al., 2006]. For materialising the said objective, researchers have developed and utilised various kinds of platforms ranging from the simple bimetallic strip used in cloth iron to relatively complex systems like based on quantum dots [He et al., 2014, Yi et al., 2013]. It is a well known fact that among the available resources for example electrical, mechanical [Pasquale, 2003] and their combination [Boland et al., 2016, Smith et al., 2013], light is the most powerful tool for sensing and measurement purposes [Fujii and Fukuchi, 2005, Fukuchi and Shiina, 2012]. Optical sensors are usually preferred because they are less costly, robust, easy to fabricate and use, moreover accurate and precise. Change in any property of the incident light for example wavelength, intensity, polarization coming from the sample under study can provide us significant information about it [Goldenstein et al., 2017, Caucheteur et al., 2016, Tao et al., 2007].

Sensing nature of light is harvested by using different experimental arrangements and geometries. Some of them to mention are optical resonators [Hu et al., 2009, Hunziker et al., 2003, Saaski et al., 1990], surface plasmon resonance [Homola et al., 1999], absorption [Tai et al., 1987, Wolthuis et al., 1992] or combination of them [Sharma and Gupta, 2004]. Optical resonators are versatile in their form and appearances. They can be spherical (micro-spheres) [Laine et al., 2001, Hanumegowda et al., 2005], cylindrical (bare optical fiber) [Eryürek et al., 2017], toroidal (micro-

toroids) [Cheema et al., 2012, Hunt et al., 2010], ring [Iqbal et al., 2010], linear (Fabry–Perot optical cavity) [Steinmetz et al., 2006, Santos et al., 1992, Corbitt et al., 2006]. This work is focused on the use of Fabry–Perot type linear resonator for the purpose of monitoring and recording the changes in the surroundings. The proposed resonator is composed of tapered single mode optical fiber and two fiber Bragg gratings, one on each side of tapered fiber. Fabry–Perot cavity was originally invented by the Charles Fabry and Alfred Perot in 1899 [Fabry, 1899, Perot and Fabry, 1899]. In its classical form FP-Cavity consists of two highly reflecting mirrors which are placed at certain distance from each other.

When light is injected from one end of the of the cavity, it begins to oscillate between the mirrors. The light beam reflected from one mirror interfere with the beam reflected from the other mirror. Depending upon the nature of their interaction, constructive and destructive interference results. All of these processes lead to the generation of standing wave pattern in the cavity [Safa, 2009]. Figure 1.1 shows a typical free space Fabry–Perot resonator comprised of mirrors “ M_1 ” and “ M_2 ”, separated by distance “ L ” and its output spectrum.

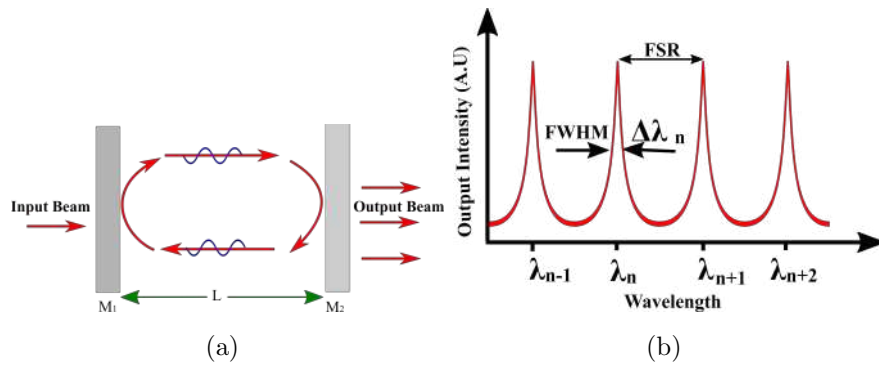


Figure 1.1: (a) A linear Fabry–Pérot resonator consisting of two mirrors, (b) an exemplary spectrum from linear resonator showing its full width half-max (FWHM) and free spectral range (FSR).

The frequency at each mode location, supported by the cavity can be determined

from the Equation 1.1.

$$v_n = n \frac{c}{2L}. \quad (1.1)$$

Here “c” is the speed of light and “n” is an integer that corresponds to mode number. The frequency of fundamental mode can be calculated by using n=1 and then Equation 1.1 gives $v_{f1}=c/2L$. If there is any loss mechanism present in the cavity, then light trapping ability of the cavity will be less. Examples of loss sources are absorbence, out of positioning of mirrors or their less reflecting power. All of these factors will contribute to the broadening of modes and reduced quality factor measured for desired mode as

$$Q = \frac{v_d}{\Delta v_d}. \quad (1.2)$$

Where “ Δv_d ” is full width at half maximum for the mode of interest. The distance between the two similar consecutive modes v_{n+1} and v_n is defined as the free spectral range (FSR) of the resonator and found by

$$FSR = \frac{c}{2n_m L}. \quad (1.3)$$

In Equation 1.3, “ n_m ” represent the refractive index of the material present into the cavity (for air $n_m=1$). So the sensors using Fabry–Perot cavity make use of different strategies for the purpose of sensing and detection. It can be change in mode location, quality factor or free spectral range with respect to external fluctuations or agent. In some instances their merger is used as well. Moreover, there are studies that achieved the desired goal of the sensing device by monitoring the variations in the variables that are extracted from Equations 1.1, 1.2 and 1.3. Alter in the Photon lifetime, absorption and phase shift are the examples of some of the most frequently used criterias.

1.1 Thesis Organisation

The thesis has been arranged in this way, chapter 1 presents some of the available optical sensing methods. Their advantages and limitations along with the leverage

of phase shift cavity ring down spectroscopy on them. It also covers the motivation, operating behind the all experimental studies. Chapter 2, will provide the information about the absorption based optical sensing methods. It starts from the simplest direct absorptive method and gradually explores the CRDS and its various forms ultimately leading to the fundamentals of PS-CRDS. Before going into the details of mathematical explanation regarding PS-CRDS, it is necessary to understand the fabrication and working of FP-cavity type linear resonator made by using FBGs and tapered optical fiber. Chapter 3, will try to develop the knowledge base about the functioning of FBGs, their fundamental concepts and detailed characterization. Functioning of normal and tapered optical fibers together with basic principles governing their behaviour will be discussed in same chapter. At the end, this chapter explains the fabrication and characterization of cavity made out of FBGs and tapered optical fiber. Chapter 4, will illuminate and explain the results obtained by the use of PS-CRDS in combination with tapered fiber-FBGs based linear optical cavity. The central point of discussion in chapter 5, is the new strategy, considered as the amplified version of simple PS-CRDS. It has the ability to improve the performance of sensor built in chapter 4. At the end of thesis, chapter 6 will highlight the conclusion and future directions of the presented research work.

1.2 Literature Review

This work is focussed on the use of fiber Bragg gratings and tapered optical fiber that has been used for making a multi-pass optical cavity. It also brings to limelight a sensing method that is based on transmission of the cavity and phase shift cavity ring down spectroscopy.

It is well known fact that, surface plasmon resonance (SPR) is a most prominent single pass optical sensing method. It is sensitive enough for many real time detection schemes and measurements. It make use of change in the location of resonance dip in the transmission spectrum as a function of detection event. Hence, in this way it eliminates the need of any kind of specific mark or label for the detection purposes. Although the initial use of SPR was limited to study the features of

thin films [Pockrand et al., 1978]. Now a days it is being used into different kinds of research fields for example life sciences, food industry, biomedical applications [Liedberg et al., 1983]. But it has some drawbacks associated with it. For example, its ability to sense heavier molecules easily as compared to lighter once [Englebienne et al., 2003]. Similarly, binding issues between ligand and analyte can also affect the sensor working [Helmerhorst et al., 2012]. Above all, the most prominent constraint on the performance of SPR based sensors is its single pass nature. That is interaction between the sample and light is just once [Catimel et al., 2001]. More accurate results can be obtained by data taken by several trials under similar conditions, which is a time consuming process. These were the shortcomings, that brought optical resonator based sensors into more and more attention for deploying in real-time sensing applications. Some other benefits are, these resonators have high Q-factor usually in the range of 10^5 to 10^{10} [Vollmer et al., 2002, Chao and Guo, 2003, Yalcin et al., 2006, Vahala, 2003] which make it possible for light to sample the analyte many times before completely dying out. In addition, these resonators have many interesting properties just few to name, change of Q-factor or resonant mode location as a function of variations in the external environment [Laine et al., 2001, Ioppolo and Ötügen, 2007, Ioppolo et al., 2011]. But using the optical resonator belonging the category of micro-resonators is not always straight forward. These are difficult to fabricate and their experimental arrangements are quite delicate.

It is also tough job to put them into resonance by finding out an optimum contact point for efficient coupling of light to them. Any kind of fluctuation which can affect that contact will result into erroneous measurements. Furthermore, various mechanisms that can be thermo-refractive in nature, laser intensity change or frequency variations during the experiment can add noise to the measurements taken by micro-resonators using resonant wavelength shift as a sensing strategy. Hence, it results in reduced performance of the device [Hu et al., 2009, Foreman et al., 2014, Matsko et al., 2007, Gorodetsky and Grudinin, 2004]. Cavity ring down spectroscopy (CRDS) is a useful detection technique that provides reasonable answer to the said problems. It was developed in 1984 [Anderson et al., 1984] and transformed

into sensing method for the first time in 1988 [O’Keefe and Deacon, 1988]. It involves the measurement of life time of photons in cavity which is independent from the noise mechanisms, e.g., laser intensity change [Berdn and Engeln, 2009, Berdn et al., 2000]. But there are some fundamental limitations associated with CRDS. This scheme is bit expensive because of the use of pulsed laser sources and fast electronics to detect and acquire the ring down time which can be of the order of 10^{-9} s [Humphries et al., 2016]. Further more, extraction of ring down time is made by the use of fitting algorithms which makes it less reasonable for real time sensing experiments [Lehmann and Huang, 2009, Romanini et al., 1997]. Although some other forms of pulsed CRDS also exist, these are for example continuous wave (CW) CRDS and cavity enhanced absorption spectroscopy (CEAS). But CW-CRDS is so much sensitive in nature. Any agent that can change the length of cavity and put it out of resonance, can severely affect its performance. On the other hand sensitivity of CEAS is effected by the laser intensity changes. Which is the figure of merit for choosing CRDS. Simple answer to the problems of CRDS is its modified form called phase shift cavity ring down spectroscopy (PS-CRDS). In PS-CRDS, intensity modulated laser beam excites the mode of the cavity, beam coming out of the cavity has different phase and depth of modulation in comparison to the input beam [Engeln et al., 1996, Bescherer et al., 2009].

This information of phase difference can be used to extract quite accurate ring down time with much smaller, simpler and economical viable arrangement [van Helden et al., 2004, Dhiman et al., 2015]. For calculating the photon life time in optical cavity, Herbelin et al. in 1980 for the first time use phase shift technique in combination with modulated light [Herbelin et al., 1980]. Later Engeln et al. in 1996 measured the transition frequencies of oxygen molecules using PS-CRDS [Engeln et al., 1996]. Performance of this methodology was further enhanced by the work of Helden et al. and Kasyutich et al. which made it insensitive to noise resulting from amplified spontaneous emission of lasers [van Helden et al., 2004, Kasyutich et al., 2006]. Initially free space optical cavities and fiber loops with lengths greater than 20m has been used for PS-CRDS demonstrations. But free space optical cavities

are not suitable for liquid phase measurements because it can affect the reflectivity of the mirrors. Moreover these arrangements are difficult to handle and operate because of their mirror alignment issues. On the other hand, for exciting the fiber loops these systems use to keep laser at fixed wavelength because free spectral range of such resonators can be smaller or comparable to laser spectral bandwidth [Tong et al., 2004]. As result sensing systems based on microresonators, e.g., microspheres or microtoroids [Barnes et al., 2013, Cheema et al., 2012] that use wavelength scan to excite individual cavity mode are superior in performance to them. But as said earlier, unfortunately placing of such resonators at the thinnest section of tapered optical fiber for the efficient coupling of evanescent field is not an easy job and sometimes can results into the breaking of tapered section because of its fragile nature [Wang et al., 2017, Vollmer and Arnold, 2008]. These drawbacks of microresonators opened the way for using linear resonators for studies. Strain sensing with the achieved sensitivity of $13.9 \text{ pm}/\mu\epsilon$ from linear resonator is presented in [Ferreira et al., 2015]. The quantification of both temperature and refractive index of the surrounding medium at the same time is another case study of using Fabry–Perot type resonators [Choi et al., 2010]. In [Rao et al., 2007], hollow core photonic crystal fiber with single mode fiber is used for the fabrication of linear cavity for the strain sensing. In the similar manner, [Tafulo et al., 2012] used hydrofluoric acid (HF) to etch optical fibers for enhancing the effect of evanescent field and subsequently utilized in Fabry–Perot cavity manufacturing.

1.3 Motivation and Objectives

Motivated by the studies that has been presented so far, objective of the current thesis can be summarised as follows

- Development of cheap, reliable optical resonator based sensor, for real time based sensing applications.
- That sensor should provide repeatable and reliable results.

- Sensor should be easy to handle, easy to install and operate with nominal maintenance cost.
- Robust in performance that is least affected by the various sources of noise, e.g., electromagnetic, weather.
- Data acquisition and its further processing should be easy without requiring any additional system for the same purpose.
- Should be able to operate equally well irrespective of measurement environments, e.g., liquid, gas.
- Should be smaller in terms of resonator size so that no need to use PDH technique or laser at fixed wavelength for developing resonant condition.
- Should be easy to fabricate.
- Geometry of the resonator should be such that coupling of light to it is an easier task.
- To show practical example of scheme for the sensing and monitoring the parameters change that has the advantages of both WGM and CRDS. But insensitive to the noise mechanisms that affect their measurements.

1.4 Optical Resonator and Sensing Methodology Utilised in Present Thesis

In this thesis linear Fabry–Pérot type resonator has been explored for the purpose of sensing. This linear resonator made out of two FBGs and tapered optical fiber. The basic purpose of tapered optical fiber is to act like a sensing probe and interaction with the surrounding environment. On the other hand, FBGs provides reflections for the cavity. This sensing arrangements is augmented by the phase shift cavity ring down spectroscopy (PS-CRDS) for the purpose of data acquisition, mathematical processing and analysis.

Chapter 2

FUNDAMENTALS OF PHASE SHIFT CAVITY RING DOWN SPECTROSCOPY

There are the wide range of systematic procedures available that are used for analysis or characterization of the sample under study. One of those methods is the study of spectrums or spectroscopy. It is used to investigate various attributes of the element of interest. It is an analytical tool that deals with the detailed examination of spectrum originating from light-matter interaction. This spectrum can be divided into two broad classes, first absorption spectrum and second emission spectrum. Absorption spectrum results when a part of incoming radiation is absorbed by the valence electrons. After this phenomenon these electrons go to higher energy states. Similarly emission spectrum originates when, these excited electrons return back to their original ground state. The whole process that give birth to either absorption or emission spectrum can be seen in Figure 2.1 below

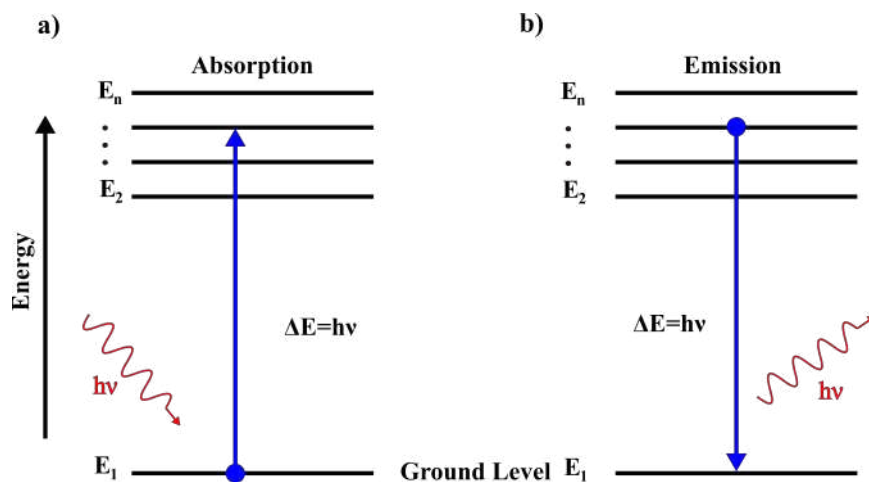


Figure 2.1: (a) An Electron excitation to higher energy state gives absorption spectrum, (b) return of electron to ground state produces emission spectrum.

The typical resultant patterns appearing after the happening of any of above said mechanism can be seen in Figure 2.2.

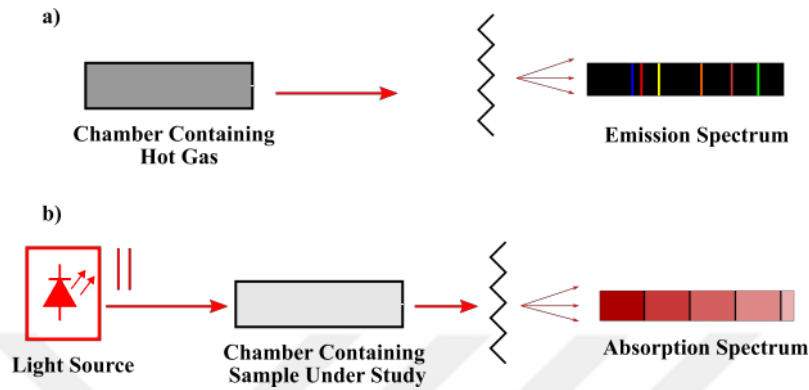


Figure 2.2: (a) Emission spectrum containing coloured lines with dark background (b) absorption spectrum with dark lines against coloured background .

Both of these spectrum kinds are being used in wide range of applications. Their use range from simple determination of the sample concentration to complex once like studying the constitution of distant heavenly bodies. Here we will focus on only those methods that deal with absorption phenomenon or spectrums arising from them. It is well known fact that, all the materials naturally have the ability to absorb electromagnetic radiations that incident on them. This property of the materials form the basis of absorption spectrums.

Absorption spectroscopy in simple words, is a method that calculates or records the absorption of incoming waves by the specimen at a particular set of wavelengths or frequencies [Gillam and Stern, 1954]. This is the tool of research, that falls into the group of most commonly used non-invasive procedures that can provide the valuable information about the sample under study without any harm, perturbation or requiring any huge volume. Absorption spectroscopy is diverse in nature. It has different forms and features that are quite distinct from each other. This differentiation can be made for example depending upon the part of electromagnetic spectrum used, it can be visible, x-rays or infra-red, or type of experimental arrangements, i.e., with or without cavity or both. Certainly cavity based approaches are superior

in performance and have higher sensitivity than non-cavity based methods. As compared to without cavity, measurement strategies using cavity are usually complex in nature. Starting with basic direct absorption spectroscopy, a non-cavity process, this chapter tries to develop a comprehensive foundation about cavity based absorption techniques like CRDS and its variants which is followed by PS-CRDS and their applications.

2.1 Direct Absorption Spectroscopy

From the Beer–Lambert law we know that, when light with intensity “ I_o ” is passed by distance “ x ” through sample with “ N ” molecules per unit volume, having an absorption cross-section “ σ ”. Then output intensity “ I ” is found as

$$I = I_o \exp(-N\sigma x). \quad (2.1)$$

The same fact is utilized in direct absorption spectroscopy to get information about the concentration or absorption cross-section of the sample under investigation. Simple experimental arrangement for direct absorption spectroscopy has been shown in Figure 2.3.

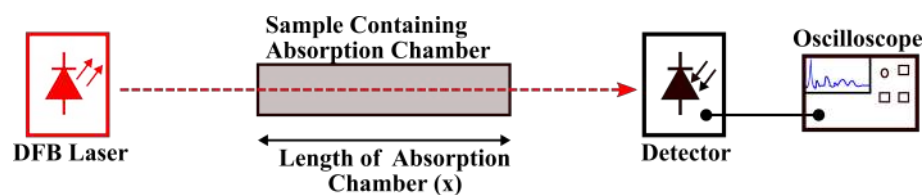


Figure 2.3: Diagrammatic representation of experimental setup used for direct absorption measurements .

This experimental setup helps to determine the light absorbed by the specimen under study. Since this absorption is dependent on the frequency of light used and composition of the specimen, then by plotting the output light intensity against frequency, absorption spectrum can be constructed. This absorption spectrum further can be used for details analysis and studies. Although there are many advantages

of direct absorption spectroscopy but it has some weaknesses as well. The most important drawbacks of this technique are

- Medium should have insignificant scattering as compared to the absorption. For the purpose of most sensitive applications scattering must be calculated and amendments should be applied for the recorded absorption.
- For the materials having so much small absorption, difficulty in measuring the minute changes in the transmitted signal on the top of large background.
- Small effective path length and its single pass nature that is light samples the analyte only once.

These limitations in turn results in its finite sensitivity and accuracy. Compared to the regular direct absorption spectroscopy there is its bit advanced and sensitive version of it, which is called wavelength modulation direct absorption spectroscopy (WM-DAS) [Du et al., 2018]. It makes use of fast Fourier transform in combination with wavelength modulated laser to find more precise and noise free absorption spectra. On the other hand, there are some methods available, that monitor the changes in the parameters of the system as the function of light absorption. These parameters can be pressure for the photo-acoustic spectroscopy or fluorescence in the case of laser induced fluorescence and ions for resonant enhanced multi-photon ionization [Berden et al., 2000]. Moreover, these experimental schemes are immune to background problems and hence superior in performance to direct absorption. But they involve complex and time consuming calibrations steps. There is the need to apply this calibration before the use of the systems, that put a constraint for their application. The proper solution to the limitation of direct absorption spectroscopy is CRDS, which is multi pass approach.

2.2 Cavity Ring Down Spectroscopy (CRDS)

CRDS is the approach in which time taken by the sample to fully absorb light is measured instead of intensity of absorbed light. First ever use of the CRDS was re-

ported to be the characterization of highly reflecting mirrors [Herbelin and McKay, 1981]. Later on, it was used by O’Keefe and Deacon for finding out absorption at molecular level [O’Keefe and Deacon, 1988]. In the short span of time since its development, it became to be recognised as highly sensitive and accurate methodology with the applications varying from simpler to complicated once, e.g., aerospace industry, reaction kinetics [Yu and Lin, 1993]. CRDS dramatically enhances the performance of device that make use of direct absorption as a main scheme. That is because of improved effective path length of the light through sample of interest. It is achieved by the use of optical cavity, which is composed of highly reflecting mirrors that provide the multiple reflections of light. If “d” is the mirrors separation, each having same reflectivity that is “ ρ_1 ” = “ ρ_2 ” = ρ then effective path length “D”, when input light intensity decreases to “1/e” of original magnitude, is given as

$$D = \frac{d}{1 - \rho}. \quad (2.2)$$

Equation 2.2 gives an effective path length of 1.0 Km for the cavity whose original length is 1 m and having mirrors reflectivity $\rho = 0.999$. Being absorption based technique in nature, it is a reasonable choice for replacing previously available absorption techniques requiring long path lengths (~ 10 km) for example differential optical absorption spectroscopy (DOAS)[Edner et al., 1993].

The principle operating behind any CRDS procedure involves the measurement of photon lifetime in high finesse optical cavity. For a typical CRDS experimental arrangement as shown in Figure 2.4(a), light from the pulsed laser source is introduced into the cavity made of highly reflecting mirrors (reflectivity $\geq 99\%$). Light travels back and forth many times between these two mirrors (M_1 and M_2) leading to series of pulsed output, as shown in Figure 2.2(a), before finally existing the cavity. The partition between two consecutive pulses or the time taken by the single pulse to complete one such cyclic tour in the absence of any lossy medium is given by

$$t_r = 2n_m \frac{L}{c}. \quad (2.3)$$

where “c” is the speed of light, “L” is the length of cavity and “ n_m ” is refractive index of medium if present otherwise $n_m = 1$. At each round trip a fraction of

light is lost that can be due to the transmission or absorption by the end mirrors and material (if any) present into the cavity. This results into exponential decay of light intensity into the cavity. By monitoring the transmission of cavity with the help of photo detector, the exponential fall in the light intensity can be observed and recorded. The relationship among photon lifetime or ring down time of cavity “ τ ”, mirror reflectivity “ R ” and absorption losses “ α ” for the sample is given as [Romanini and Lehmann, 1993, Yu and Lin, 1993]

$$\tau = \frac{t_r}{2[(1 - R) + \alpha l_s]} \quad (2.4)$$

In Equation 2.4 “ l_s ” is the effective path length. In the absence of any absorptive analyte that is $2\alpha l_s = 0$, for the cavity with the length ranging from 0.1 to 1 m and having highly reflective mirrors, photon lifetime upto microseconds can be achieved. But in the presence of lossy medium ring down time gets significantly reduced as governed by Equation 2.4 and can be seen in Figure 2.4(b).

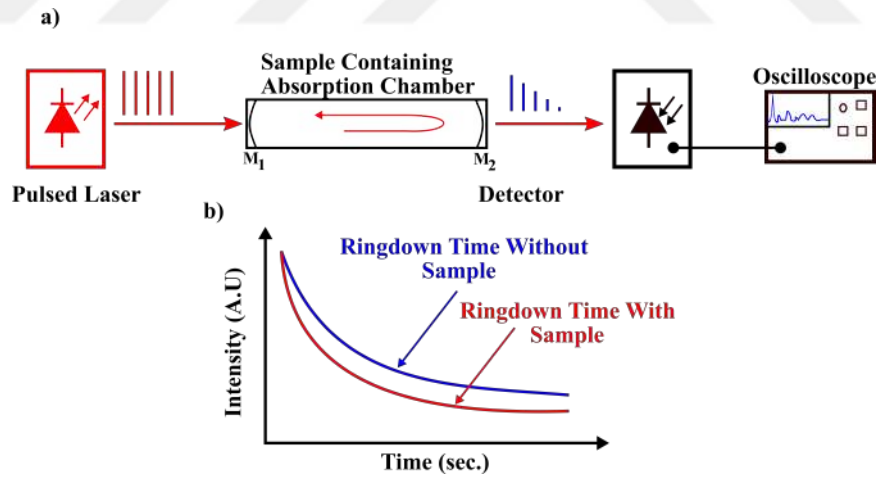


Figure 2.4: (a) An illustration of experimental arrangement utilized for cavity ring down spectroscopy, (b) ring down time variation with and without sample.

Difference between the two ring down times serve as an indicator for the detection and sensing. For the highly absorptive materials, path length will be small and corresponding ring down time will be less. But for lesser absorptive environment both of these variables will have significantly higher values. This means increased

sampling which shows up in the form of higher sensitivity and accuracy for lesser absorptive materials. The most obvious benefit of CRDS is that, there is no effect of change in the amplitude or the pulse profile of the laser on the measurements taken by CRDS [Sattler, 2020]. It is because photon lifetime is used to find material absorption as compared to the absolute value of light intensity available into the cavity. An other edge of CRDS is its use to find the absorption coefficient “ α ”. Let “ τ ” and “ τ_s ” are the ring down times without and without sample receptively and assume refractive index of the sample be “ n_s ” then

$$\alpha = \frac{1}{c} \left(\frac{n_s}{\tau_s} - \frac{1}{\tau} \right). \quad (2.5)$$

Equation 2.5 accurately calculates the absorption coefficient “ α ” (cm^{-1}) and hence eliminating the requirement of calibration process.

2.3 Variations of CRDS

There are different experimental designs for performing CRDS. The selection of each CRDS scheme is decided by the nature of application and final desired sensitivity to reach. These various ways for implementing CRDS differs from each other in the type of light source, e.g., continuous wave [Simpson, 2003, He and Orr, 2006], cavity used, e.g., fiber based [Sharma et al., 2018, Waechter et al., 2010], free space [Berden et al., 2000] [Tong et al., 2004, Reid et al., 2011] and experimental configuration that is presence or absence of feedback [Butler et al., 2009]. The short description of some of the most frequently applied variations of CRDS has been given at this moment

2.4 Continuous Wave CRDS (CW-CRDS)

The classical method of CRDS make use of pulsed laser source. But because of their comparatively large bandwidth (BW) and ability to excite more than one modes of the optical resonator, these systems usually have less sensitivity. The performance of classical CRDS systems can be improved further by incorporating continuous wave (CW) laser along with beam modulator. One of the characteristic features

of CW-laser is that they usually have BW lesser than 10 MHz. Experimental layout used for CW-CRDS has been shown in Figure 2.5. In the literature there are many studies available that present the use of CW-CRDS for making analysis and detection. Some of them to mention are, [Macko et al., 2004] and [Liu et al., 2007] reported to used CW-CRDS for calculating water vapours and methane absorption, respectively. In the same manner [Dudek et al., 2003] showed moisture sensing and [Simpson, 2003] dinitrogen pentoxide concentration monitoring with the similar setup. Moreover, [Tarsa et al., 2004a] used tapered optical fiber and CW-CRDS pair for loss determination.

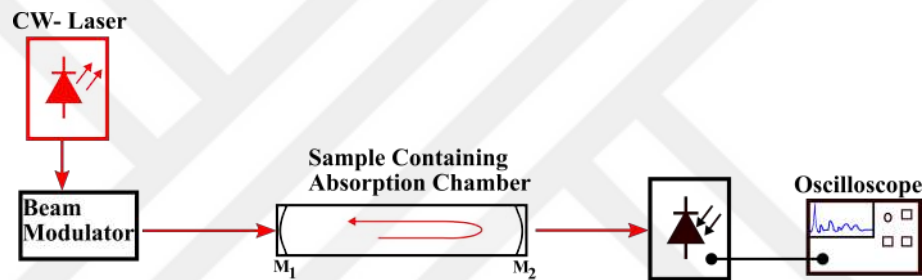


Figure 2.5: An experimental setup for continuous wave cavity ring down spectroscopy with beam modulator as its necessary component.

But CW-CRDS systems are so much delicate in nature. Any parameter, e.g., thermal, mechanical that can affect the length of the cavity can bring about huge changes in the output data. Which will end up into more noise and reduced sensitivity [Simpson, 2003] of the device.

2.5 Cavity Enhanced Absorption Spectroscopy (CEAS)

Cavity enhanced absorption spectroscopy relies on the decrease in input light intensity. This reduction in light intensity is attributed to escape of light from the mirrors and light absorption by the sample or mirror surface. Experimental details of CEAS is shown in Figure 2.6. Light intensity inside the cavity and ring down time are related to each other [Engeln et al., 1998]. In this way absorption measurements can be made from intensity profile. Let “ I_{in} ” and “ I_{out} ” symbolize

the input and output light intensities, respectively for the cavity. It is important to observe that no modulation is applied to the input light intensity “ I_{in} ”. Hence for CEAS no modulator is required.

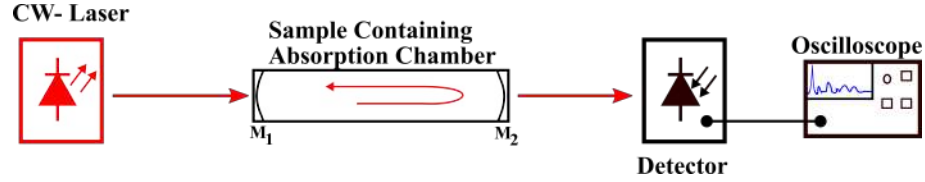


Figure 2.6: Depiction of configuration used for performing cavity enhanced absorption spectroscopy.

Suppose that the said cavity is composed of two mirrors with same reflectivity given by “ ρ ” and distance between them is “ d ”. Then absorption is calculated as [Ball et al., 2004].

$$Absorption = \left(\frac{I_{out}}{I_{in}} - 1 \right) \left(\frac{1 - \rho}{d} \right). \quad (2.6)$$

In other words CEAS records time-averaged output of the cavity for sensing. In addition to other dissimilarities it is different from CW-CRDS in that because it does not require beam modulator. Although CEAS is easier to implement than that of CRDS but it is less sensitive compared to the latter. It is because of the affect of laser intensity changes to which time domain approach, i.e., CRDS is exempted. But its worth mentioning that CRDS and its variants discussed so far require an expensive and fast data acquisition systems to measure the ring down time, which can be as small as nano seconds.

Moreover curve fittings are performed for calculating ring down time [Berden and Engeln, 2009]. That involves the addition of noise term in the final result and hence limiting the use of CRDS for analysis requiring more sensitivity. By considering these facts it is easy to say that phase shift cavity ring down spectroscopy (PS-CRDS) is superior. PS-CRDS is another variation of simple CRDS. It does not require costly equipment for data acquisition or for pulse generation and ring down time can be found easily either from phase shift or modulation depth. PS-CRDS

has the similar sensitivity or in some cases even higher than its counter parts like simple CRDS or its variants [Tong et al., 2004].

2.6 Phase Shift Cavity Ring Down Spectroscopy (PS-CRDS)

PS-CRDS is a technique in which intensity modulated light excites the modes in optical cavity. The light beam recorded at the output end of the cavity has similar intensity of modulation but entirely different magnitude of phase and modulation depth compared to the original input beam [Cheema et al., 2012, Tong et al., 2004, Reid et al., 2011]. For the sake of understanding the concept of PS-CRDS, let us consider a simple case in which linear cavity is made of two end to end connected/spliced FBGs. This cavity has been put into the resonance condition with the help of intensity modulated laser coming from the DFB laser. Tuning of the laser to the central peak reflecting wavelength of the FBGs is done with the help of temperature and current settings of laser controller. Proposed cavity has been shown diagrammatically in Figure 2.7.

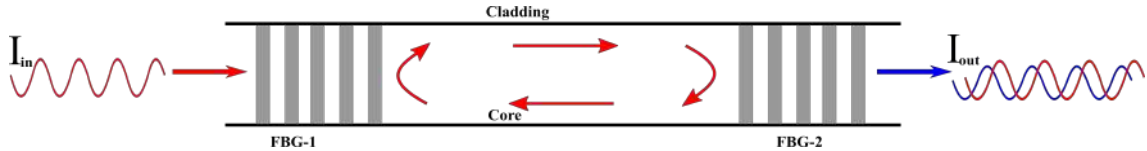


Figure 2.7: A FBGs based linear optical cavity with intensity modulated input and output laser beam.

Quality factor “Q-factor” for the linear resonator of unit length is given as

$$Q = \frac{2\pi c\tau}{\lambda_r}. \quad (2.7)$$

Where “ λ_r ” and “ c ” are the resonant wavelength for the cavity and speed of light, respectively. Consider “ I_{in} ” is the intensity modulated light input to cavity, which is given by the Equation 2.5.

$$I_{in} = I_o[1 + \alpha \sin(\omega t)]. \quad (2.8)$$

where “ α ” is the modulation depth and “ $\omega = 2\pi f$ ” is the angular modulation frequency of the input intensity. The impulse response “ h ” of the cavity can be given as

$$h = C \exp\left(\frac{-t}{\tau}\right). \quad (2.9)$$

Where $C = (1/\tau)$ by the law of conservation of energy. Let “ I_2 ” is the intensity of coupled light. Then intensity of modulated light “ I_{cav} ” launched into the cavity is given by

$$I_{\text{cav}} = I_2[1 + \alpha \sin(\omega t)]. \quad (2.10)$$

The output intensity from the cavity is obtained by convolving cavity impulse response with the light intensity coupled into the cavity [Engeln et al., 1996, Reid et al., 2011] as follow

$$I_{\text{out}} = \frac{1}{\tau} \int_{-\infty}^t I_2[1 + \alpha \sin(\omega t')] \exp\left[\frac{-t + t'}{\tau}\right] dt'. \quad (2.11)$$

by simplifying further we get

$$I_{\text{out}} = I_2 \left\{ 1 + \frac{\alpha}{\sqrt{1 + (\omega\tau)^2}} \sin(\omega t - \arctan(\omega\tau)) \right\}. \quad (2.12)$$

From Equation 2.12 phase shift “ ϕ ” between input and output of the cavity is related to ring down time “ τ ” by expression

$$\phi = -\tan^{-1}(\omega\tau). \quad (2.13)$$

Negative sign in Equation 2.13 shows output lags compared to input signal. From Equations 2.4 and 2.13, it is apparent that anything that will affect the ring down time or Q-factor will also affect phase and subsequently can be sensed or detected. This effect can be observed easily in Figure 4.4(a) with the increase of losses of cavity. It is important to note that modulation depth of the output “ α' ” and input intensity will not be same and relation between them is given by the expression

$$\alpha' = \frac{\alpha}{\sqrt{1 + (\omega\tau)^2}}. \quad (2.14)$$

As said earlier, it is possible to calculate the ring down time from Equation 2.4 or Equation 2.6 hence eliminating the needs for fast and expensive data acquisition systems. Moreover, information about the phase shift alone, can provide the limit of detection, sensitivity and response time that one can get for the CRDS [Tong et al., 2004] or its variants.



Chapter 3

FABRICATION AND CHARACTERIZATION OF FIBER BRAGG GRATING BASED LINEAR CAVITY

Optical cavities come into various forms and shapes ranging from free space ones to the spherical or toroidal cavities. Basic purpose of any type of optical cavity is to provide confinement of light. Optical cavity that has been proposed in this thesis is linear in nature that is Fabry-Perot type. For achieving the light oscillations (confinement) within the cavity and its role as a sensing probe, it makes use of two principal components which are

- Fiber Bragg gratings (FBGs).
- Tapered single mode optical fiber (T-SMF).

Each of the above said element has its own individual characteristics. It depends upon the conditions and material used for their fabrication. Their individual behaviours add up and observed as overall working of the single unit that is linear cavity. FBGs find their foundation in Bragg mirrors. It is difficult to understand their functioning, e.g., wavelength selection, filtering without the basic knowledge about the Bragg mirrors. This chapter discuss FBGs and T-SMF in detail before finally leading to their unification in the form of linear cavity and its performance analysis.

3.1 Bragg Mirror or Reflector

Mirrors find many applications in our daily routine. Their use vary from simpler as seeing oneself during combing, traffic behind on road to much complex once like in observatories, controlling light for viewing minor hidden details in biological samples

etc. Technically speaking mirror is a reflective shiny plane which directs incoming light in different directions. This function is performed in away that incoming and reflected light subtend same angle on the plane. The ordinary mirrors used to have a layer of silver or chromium that carry out the job of reflection. But for areas like lasers, photonics, optical cavities that involve the precise selection of wavelength of interest and higher reflectivity ($\geq 99\%$) these mirrors are not the first choice any more. For this goal to achieve another specialized class of mirrors are used. These mirrors are called dielectric or Bragg mirrors. They are formed by depositing the layers of dielectric medium, one after the other, having low " n_l " and high " n_h " index of refraction. One of the such mirrors is shown in Figure 3.1.

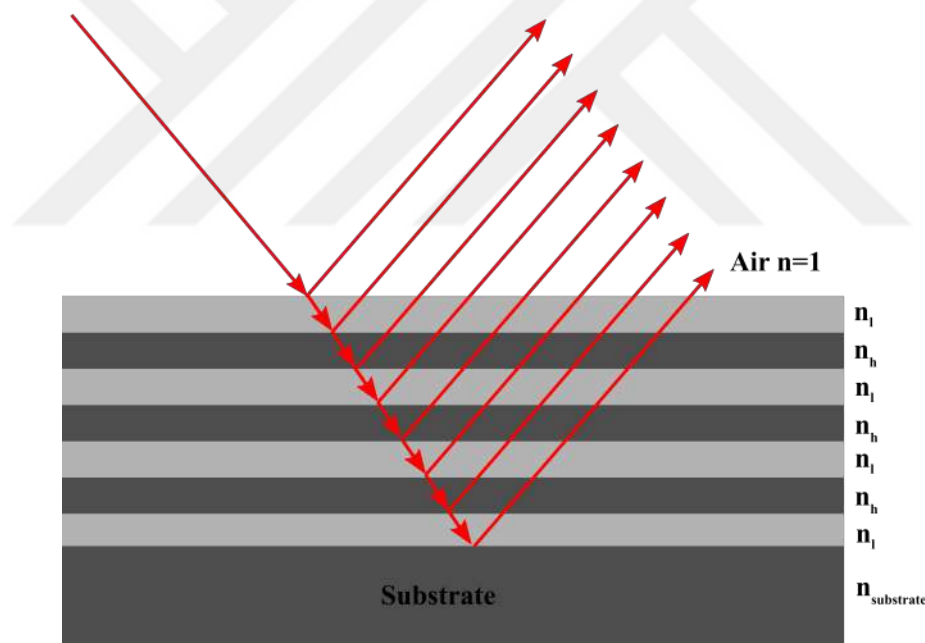


Figure 3.1: A dielectric mirror with alternative high and low refractive index layers. Each successive layer reflects back some portion of incident light.

For the central wavelength of λ_c , width of the layers can be found from the relations $w_h = \lambda_c/4n_h$ and $w_l = \lambda_c/4n_l$, respectively. After the incidence of incoming ray of light on the surface of dielectric mirror, some part it get reflected at each low-high index boundary. Each of these reflected parts of incoming light have 0° or multiple of 360° phase difference with respect to each other. Hence constructive interference

results. So, with the passage of time, reflected light becomes stronger and stronger but on the other hand incoming wave becomes weaker and weaker. Provided that, absorbance by the coatings is ignorable. Refractive indices of the layers and FWHM of the maximum reflectance region is related by the following Equation 3.1.

$$\Delta\lambda = \lambda_c \frac{4}{\pi} \sin^{-1} \left[\frac{n_h - n_l}{n_h + n_l} \right]. \quad (3.1)$$

Selection of the dielectric mirror for a specific use depends upon many factors. Some of them are

- Desired **wavelength** at which mirror will be operated.
- Depending upon the application, their **geometry** can be curved, plane or any other form.
- For some devices just 90% **reflectivity** is enough. But some other require reflectivity 99% or even higher like 99.99% for example lasers, telescopes. Highly reflective dielectric mirrors are usually costly due to the expensive manufacturing processes.
- Since their reflectivity changes with the **angle of incoming ray** angle of incoming ray, so they have different reflectivity for different angles. For example a mirror with reflectivity of 99.99% made for 30° will not have same reflectivity for 70°.
- The **environment** where they will be deployed. This parameter directly influence the material that will be used for mirror fabrication.

3.2 Fiber Bragg Gratings (FBGs)

Past three decades has undoubtedly seen a tremendous development in the field of fiber optic technology. Use of optical fibers in the fields of sensors, mechanical inspection, telecommunications [Liu and Tam, 2016, Giles, 1997] is increasing with the passage of time. Recently researchers got the ability to bring periodic variation

in the refractive index of single mode optical fiber (SMF) core [Hill et al., 1978, Kawasaki et al., 1978]. This feat was achieved by exposing the germania doped fiber to the Argon-ion laser. Photosensitivity of germania doped fiber is held responsible for the change in the RI of core. For the wavelength, fulfilling the Bragg condition, these RI variations behave like a mirror or we can say that fiber Bragg gratings have been formed. Reflectivity and working wavelength of FBG is determined by many factors for example length of its grating pattern, period and vigorous changes into RI of the core [Frazão et al., 2005].

3.3 Manufacturing of The Fiber Bragg Gratings

Core of the optical fibers show sensitivity to UV light. It is because, they are doped with Germanium for having relatively higher RI compared to the cladding. In the context of photo-sensitivity of core implies that, when UV light is shinned on the core, it brings about irreversible variation in its RI. This effect can be amplified further by the addition of hydrogen. In the typical process for the fabrication of FBGs shown in Figure 3.2. UV light from excimer laser is passed through phase mask. This results in interference pattern to which photosensitive fiber is exposed.

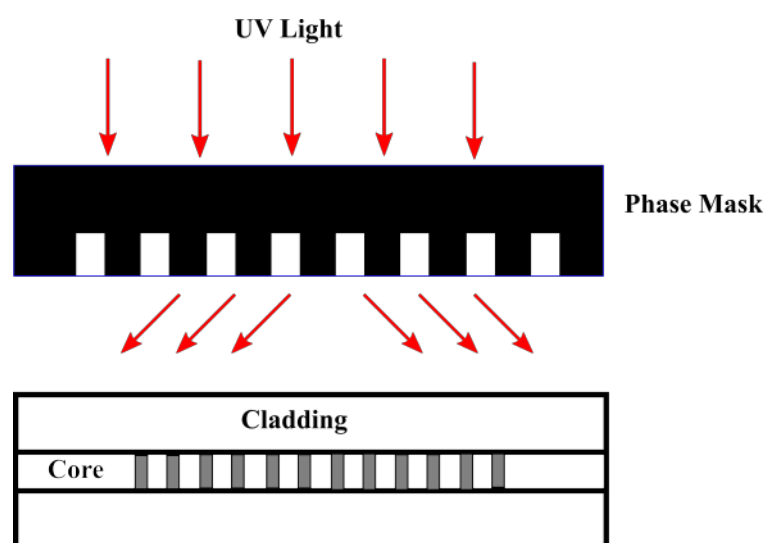


Figure 3.2: UV light through phase mask produces the refractive index modulation.

The periodically variation of light and dark bands of interference pattern creates similar zones of permanent RI change on the core of optical fiber.

3.4 Working of The Fiber Bragg Gratings

Let us consider “ λ_i ” is the wavelength incident to the typical FBG as shown in the Figure 3.3 below. Corresponding reflected and transmitted wavelengths are “ λ_B ” and “ $\lambda_i - \lambda_B$ ”, respectively.



Figure 3.3: An exemplary FBG that reflects part of wavelength that satisfies Bragg condition.

If for this FBG “ n_{eff} ” is the effective RI of the optical fiber core and “ Λ ” being its grating period then Bragg wavelength “ λ_b ” is given by

$$\lambda_b = 2n_{\text{eff}} \Lambda . \quad (3.2)$$

So only that wavelength will be reflected (Bragg wavelength) back which will satisfy the Equation 3.2. While the other wavelengths will appear at the output.

3.5 Measurement of Transmission Spectrum

Experimental arrangement for the observation of transmission spectrum of the FBG is shown in Figure 3.4(a). For the purpose of seeing transmission spectrum a broad beam light source is required. Booster optical amplifier is well known broad beam light source. For our case, light from the booster optical amplifier (BOA), is directed to FBG with the help of optical circulator. Main function of optical circulator here is to provide an isolation. Hence it avoids any kind back reflection of light in to the BOA. These back reflections in addition to adding noise into the recorded data, can

damage the performance of the optical instruments, e.g., laser [Budzyn and Rzepka, 2008, Moon et al., 2007]. So, resultant light beam, transmitted by the FBG is analysed with the help of optical spectrum analyser (OSA). Keeping in view the forthcoming experimentations, transmission spectrum of two ready made FBGs has been observed and shown in Figure 3.4(b) and (c). For the ease of recognition these FBGs have been named as FBG-A and FBG-B. In both of the transmission spectrums, a notch is visible. This notch in the transmission spectrum shows the part of the input wavelength that fulfils the Bragg condition given in Equation 3.2 and subsequently reflected back by the FBG.

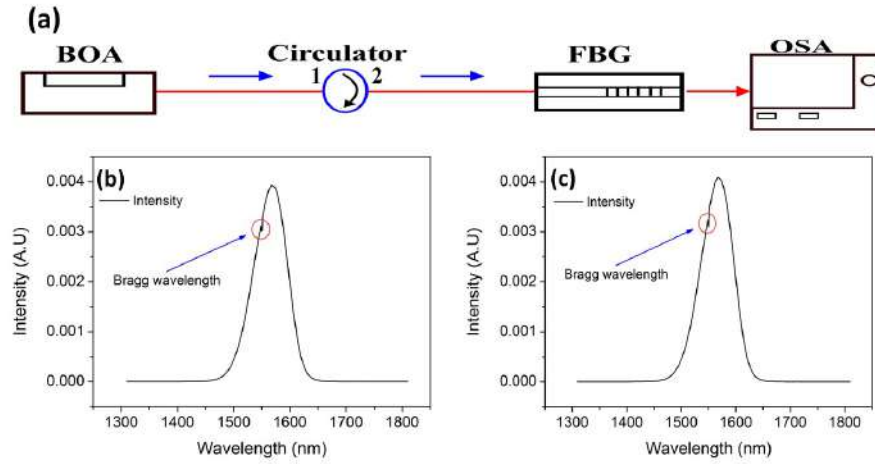


Figure 3.4: (a) An experimental layout for transmission spectrum measurement, (b) transmission spectrum of FBG-A, (c) transmission spectrum of FBG-B.

Lorentzian fittings was applied to each of the recorded transmission spectras of FBGs to determine FWHM. It came out to be 61.09 nm for FBG-A and 61.01 nm for FBG-B.

After finding out the transmission spectral response for each of the two FBGs, next step was to observe collective transmission spectrum of them. For this purpose, two FBGs was connected together end-to-end with the help of single mode simplex adapter and recorded the transmission spectrum. Experimental layout for observing the spectral behaviour for the pair of FBGs is shown in Figure 3.5(a) and 3.5(b).

Again we can see notch in the transmission spectrum of the two FBGs connected together, which shows part of the input signal that has been reflected back. Lorentzian fitting of the transmission spectrum gave us FWHM of around 60.8 nm.

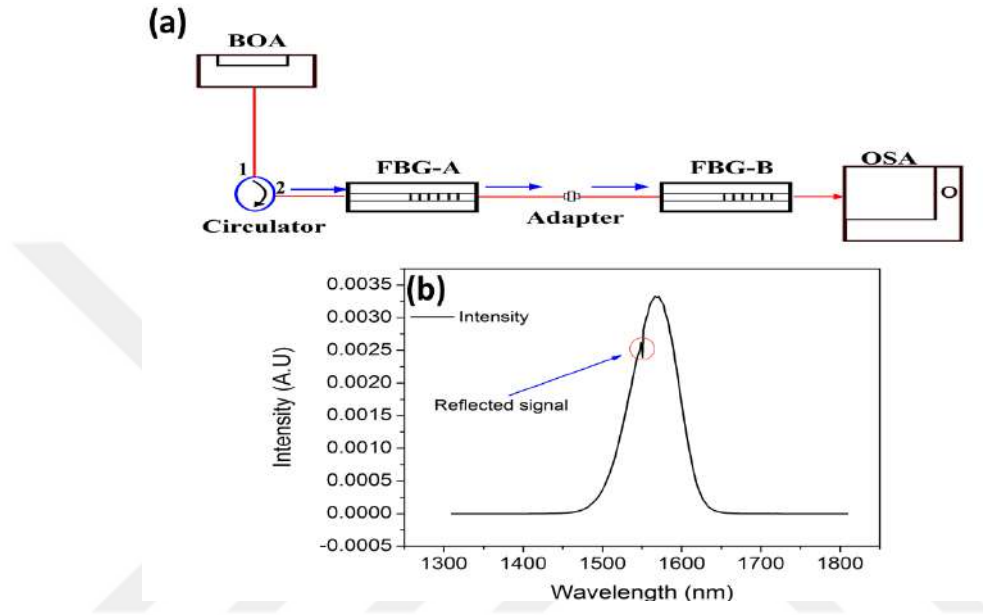


Figure 3.5: (a) Diagrammatic depiction of transmission spectrum measurement, (b) transmission spectrum of two FBGs connected end-to-end.

It is quite visible from Figure 3.4(a) and 3.5(a) that experimental configuration for recording transmission spectrum in both cases are same.

3.6 Recording of Reflection Spectrum

Measurement of the reflection spectrums for the FBGs is a bit tricky process. It involves the use of non-reciprocal nature of optical circulator. Optical circulator is a device that transmits light in one direction only. In simple words, signal applied at its port-1, appears at upcoming consecutive port-2 with almost same intensity. This transmission from one port to other port takes places without any back reflection to earlier ports. The corresponding reflected signal appears at the very next port-3. Experimental arrangement shown in Figure 3.6(a) make use of this fact to analyse the reflection spectrums of FBG-A and FBG-B. The recorded measurements for the

reflection spectrum are shown in Figure 3.6(b) and (c). As in the case of transmission spectrum, FWHM for the reflection spectrums of both FBGs calculated by making Lorentzian fitting. So FWHM, for FBG-A is 0.150 nm and FBG-B is 0.162 nm. The corresponding central wavelengths “ λ_c ” for FBG-A and FBG-B found to be 1549.87 nm and 1549.9 nm, respectively.

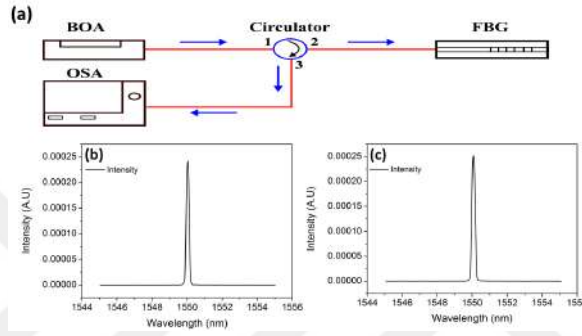


Figure 3.6: (a) Configuration for observation of reflection spectrum (b) reflection spectrum of FBG-A, (c) reflection spectrum of FBG-B.

In the last step, reflection spectrum for the FBG-A and FBG-B connected end to end was recorded and measured for FWHM which is equal to .167 nm. Figure 3.7(a) and (b) shows the set-up and recorded spectrum, respectively.

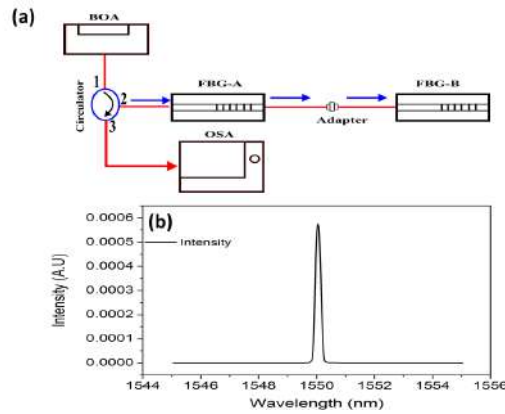


Figure 3.7: (a) An experimental arrangement for the study of reflection spectrum (b) reflection spectrum of two FBGs connected end-to-end.

The measurement of reflection spectrum of two FBGs connected end to end is important. It is because of the reason that, it helps to tune the wavelength of laser at or close to this central wavelength for the cavity build up.

3.7 Optical Fiber

A geometrically cylindrical wave guide and made of dielectric material is called optical fiber [Snyder and Love, 2012, Okoshi, 2012]. Materials used for the manufacturing of optical fibers are usually glass and plastic [Paek, 1999, Zubia and Arrue, 2001]. Light signal travelling through the optical fiber suffers losses depending upon the class of optical fiber and wavelength used for transmission [Ma et al., 2015]. Systems deploying fiber optics are superior in performance because of many reasons. Some of them are [Lee, 2003]

- Small in size (diameter) and lesser in weight.
- Neither they are affected from electromagnetic interference (EMI) nor they cause any such problems.
- Signal encounters lesser loss.
- Less susceptible to environmental changes and damage.
- Ease of installation, long life span and less maintenance cost.
- Large bandwidth and ease of signal transmission.

3.8 Construction and Principle of Operation

Each optical fiber has three distinctive components named as core, cladding and protective jacket. Core and cladding are made of the same material but refractive index of the core " n_{co} " is bit higher than that of cladding " n_{cl} ". Light travels into the core of the optical fiber because of total internal reflection (TIR). When light with angle of incidence greater than the critical angle " θ_c " strikes at the core

cladding interface gets total internally reflected. Critical angle “ θ_c ” for the core-cladding interface is given by

$$\theta_c = \sin^{-1}\left[\frac{n_{cl}}{n_{co}}\right]. \quad (3.3)$$

Figure 3.8 shows an exemplary optical fiber and total internal reflection mechanism taking place in it.

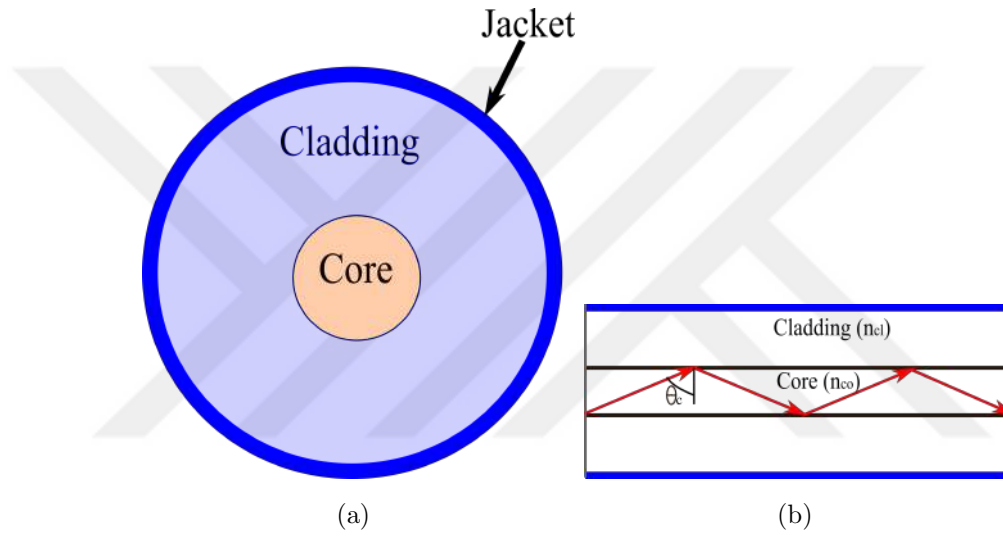


Figure 3.8: (a) An optical fiber with three distinct sections, (b) mechanism of total internal reflection in an optical fiber .

Light escapes the optical fiber and can not be confined for any value of incidence angle lesser than the critical angle “ θ_c ”.

3.9 Single and Multi-Mode Optical Fiber

Light during its travel through an optical fiber experiences a phase shift at each core-cladding interface. In a typical optical fiber, there are limited number of distinct optical paths that result in constructive phase shifts and hence strengthen the transmission. These paths are comparable to electromagnetic modes. Since different modes are incident on the fiber axis at different angles, so they have different path lengths along the fiber. Only fundamental zero-order mode travels along the core of

the optical fiber without undergoing reflection from core-cladding interface. Optical fiber supporting only fundamental zero order mode is called single mode optical fiber (SMF). It has core diameter between 8 to 10 μm . But multi-mode fibers (MMF) can handle many of electromagnetic modes. MMF typically have core diameter greater than 62.5 μm . In addition to wavelength used, the number of modes that can travel through an optical fiber, are also dependent on the diameter and refractive index of the core. The relation between them is given by the Equation 3.3 below

$$V = \frac{2\pi a}{\lambda} \sqrt{n_{\text{co}}^2 - n_{\text{cl}}^2}. \quad (3.4)$$

Where “a” is the radius of the core and “ λ ” is the wavelength of light used. If the value of “V” is lesser than 2.405 then optical fiber will be single mode otherwise it will support more than one mode, i.e., multi-mode fiber.

3.10 Evanescent Field of Optical Fiber

Now a days in addition to telecommunication, use of optical fibers is increasing in the field of sensing technology [Pitt et al., 1985, Bartlett et al., 2000]. It is a well known fact that, evanescent field (EVF) results, when electromagnetic field of the mode propagating through the optical fiber extends across the core-cladding boundary as shown in Figure 3.9.

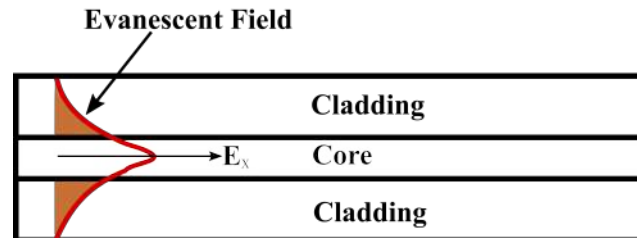


Figure 3.9: Propagation of an evanescent field in the core of optical fiber.

But problem is that this evanescent field shows an exponential decay with the in-

crease of distance “ x ” from interface and is given as

$$E(x) = E_o \exp\left(\frac{-x}{d_p}\right). \quad (3.5)$$

Where “ d_p ” is the depth of penetration calculated as [Gupta et al., 1996, Ahmad and Hench, 2005]

$$d_p = \frac{\lambda}{2\pi \sqrt{(n_{co} \sin \theta)^2 - (n_{cl})^2}}. \quad (3.6)$$

“ θ ” and “ λ ” are the angle of incidence and wavelength, respectively. Tapering of the optical fiber is one of the methods by which intensity of evanescent field can be increased into the surrounding environment. Tapering of optical fiber results in reduced V-number [Ding et al., 2005] and surroundings starts serving as new cladding [Frazao et al., 2008] and new penetration can be found by

$$d_p = \frac{\lambda}{2\pi n_{co} \sqrt{(\sin \theta)^2 - \left(\frac{n_{cl}}{n_{co}}\right)^2}}. \quad (3.7)$$

replacing “ n_{cl} ” with surrounding RI “ n_{su} ” in Equation 3.6. Optical fiber sensors based on the interaction of evanescent field with the surrounding environment are being used for sensing gases, contamination, refractive index, humidity, temperature, pH [Gaston et al., 2003, De-Jun et al., 2014], biomedical applications [Preejith et al., 2003, Liu and Tan, 1999]. For the typical SMF, evanescent field is not that much strong but its strength and resultant interaction with the analyte under observation can be enhanced significantly [Bobb et al., 1990, Ogert et al., 1992] by tapering the fiber [Mignani et al., 1998].

3.11 Tapered Optical Fiber

This section explains the tapering of single mode optical fiber. In the preparatory phase for tapering, protective jacket of the optical fiber is removed with the help of mechanical stripper and fiber is cleaned by using isopropyl alcohol (IPA). After that, tapering of the optical fiber is done by simultaneously heating and axial pulling the optical fiber with the help of motorized stages. Different kinds of heating sources

can be used for heating the optical fiber during the tapering process some of them to mention are heating torch, electrodes [Kieu and Mansuripur, 2006, Corres et al., 2006]. An exemplary tapering setup consisting of hydrogen torch and computer controlled motorized stages has been shown in Figure 3.10.

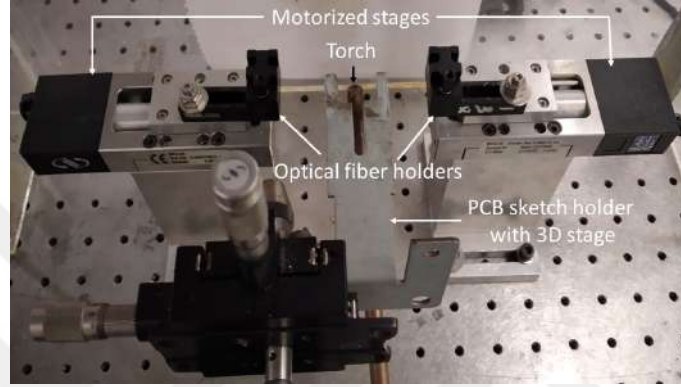


Figure 3.10: An exemplary tapering arrangement with its necessary components.

Relation between the taper length “ L_o ” and waist diameter “ $2r_w$ ” with the axial extension length “ x ” is given by [Kenny et al., 1991] the Equation 3.8.

$$r_w(x) = r_o \exp^{\frac{-x}{2L_o}}. \quad (3.8)$$

Note “ r_o ” is the initial radius of the fiber, i.e., before tapering. A typical tapered optical fiber consists of thin taper waist with a transition region on each side as shown in Figure 3.11.

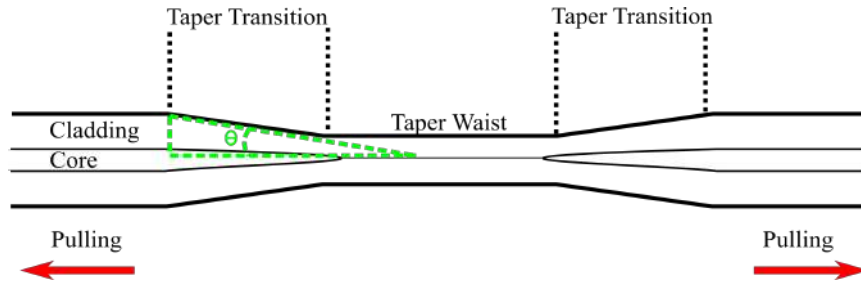


Figure 3.11: A tapered optical fiber, marking the taper angle and transition regions

Based on the parameters used for tapering, e.g., temperature, speed of motorized stages, pulling distance one can manufacture tapers having desirable properties, geometry and taper angle " θ ". Depending upon " θ ", generally tapered optical fiber can be of two kinds adiabatic and non-adiabatic [Zibaii et al., 2010, Yerolatsitis et al., 2014, Hale et al., 1996, Ji et al., 2013]. Geometrical structure of adiabatic and non-adiabatic taper can be seen Figure 3.12. It is quite apparent that " θ " for adiabatic taper is smaller than that for non-adiabatic taper.



Figure 3.12: Variation of taper angle for both: (a) adiabatic taper, (b) non adiabatic taper .

In adiabatic tapers there is no coupling of power from the fundamental mode (LP_{01}) to higher order modes and hence significant part of the input optical power remains with the fundamental mode. Necessary and sufficient condition for the adiabatic taper is that radius of the taper should change in small steps [Lacroix et al., 1988, Gonthier et al., 1987] or in other words it should have small taper angle as can be seen in Figure 3.12.

3.12 Construction Linear Cavity

Manufacturing of the linear cavity starts with the tapering of single mode optical fiber (SMF-28). It is done by first removing the protective coating of the fiber with the help of mechanical stripper and cleaning it with the wiper dipped into the isopropyl alcohol. In the next step fiber is clamped into the fiber holders provided on the computer controlled motorized stages as shown in Figure 3.10. After that, optical fiber is heated with the help of hydrogen flame while pulling at the same

time with the velocity 0.2 mms^{-1} and acceleration of 2 mms^{-2} to achieve reasonable thickness for purpose of optimal sensing. At the end of pulling, an additional pulling of 0.1 mm to 0.3 mm is also applied to avoid any kind of possible sagging or to straighten the tapered region of optical fiber. This extra pulling also ensures steady operation of the fiber during the experimentation. The resultant tapered fiber is now fixed on the mould derived from the piece of printed circuit board (PCB) as shown in the Figure 3.13(b) with the help of epoxy glue. Settling time of about 10 to 15 minutes is usually required for the epoxy glue to bind optical fiber on the PCB mould properly

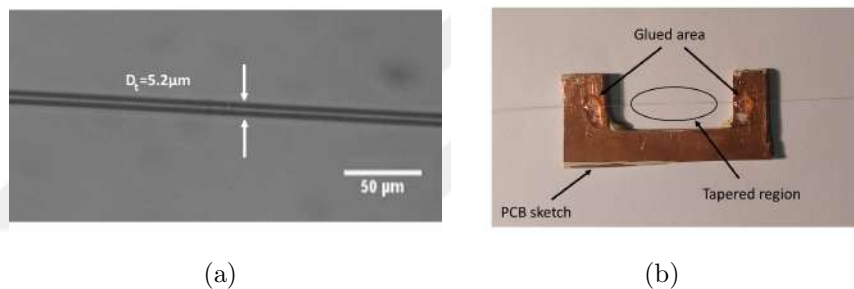


Figure 3.13: (a) Tapered optical fiber, (b) an optical fiber attached to PCB mould with glue for steady operation.

Last step of the cavity fabrication involves the splicing of tapered section with the pigtails (FC/APC 9/125 single mode), one on each side, with the help of fusion splicer. Before splicing the pigtails with the tapered optical fiber, it is important to prepare them for the splicing. It involves removing the protective jackets and coatings of optical fiber followed by cleaning with iso-propyl alcohol (IPA). Cleaning with iso-propyl alcohol (IPA) is required to make sure fiber is clean and it avoid any damage to blade of fiber cleaver. After cleaning, these optical fiber sections are cleaved. Cleaving of optical fiber sections are important in order to have flat end faces which are at right angle (90°) to axis of the optical fiber. For performing the cleaving, mechanical cleaver is used that cleaves the optical fibers. The prepared parts of optical fibers are placed in to the magnetic chucks provided on the splicer.

After that fusion splicer automatically splice the fiber by following predefined sub-routines. Fusion splicer used for splicing is shown in Figure 3.14.

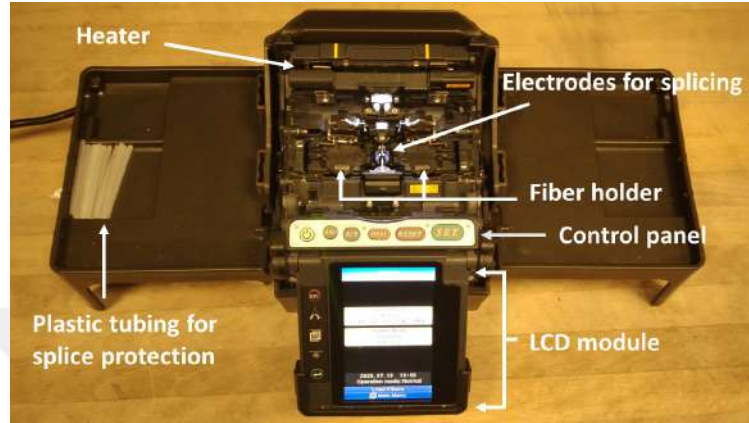


Figure 3.14: Fusion splicer.

Subsequently, adapters (FC/APC single-mode simplex) are used for connecting the whole spliced assembly with the FBGs. The spliced pigtails and tapered optical fiber has been shown in Figure 3.15.

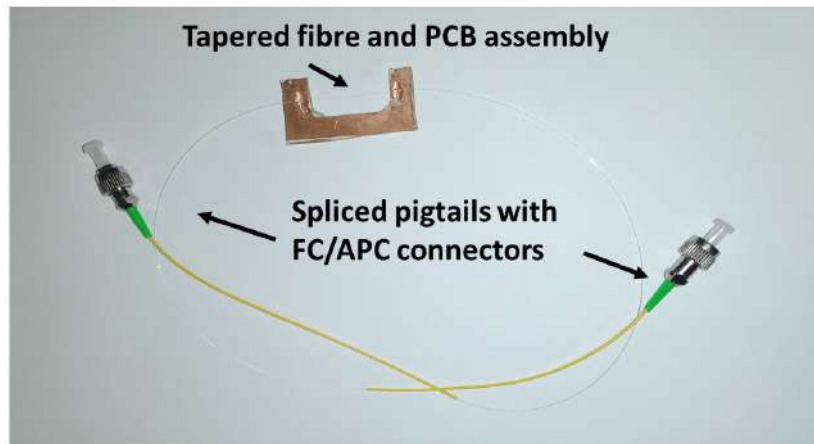


Figure 3.15: Tapered fiber and pigtail assembly .

After the connecting with FBGs, cavity containing tapered fiber is formed that can be used for sensing and further analysis.

3.13 Working of The Cavity

The linear cavity, whose fabrication has been described in previous sections, is made of tapered optical fiber and two FBGs, one on each side of the tapered fiber. Tapered optical fiber and FBGs are joined with the help of adapter (FC/APC single-mode simplex). When such a cavity is excited with the help of tunable laser, these gratings act as mirror and provide the oscillations of light. The oscillating light in the cavity shows up as a typical repeated peak pattern. For this resonance condition to achieve, it is necessary that DFB laser diode should be tuned at the wavelength matching the central wavelength of the gratings. Laser diode controller is used to tune the laser to central wavelength either by changing the injection current or temperature of the diode. The distance between the two consecutive peaks of similar nature is called free spectral rang(FSR) which is about 0.2459 pm for this scenario. Experimental arrangement and transmission of cavity can be seen in Figure 3.16(a) and (b), respectively.

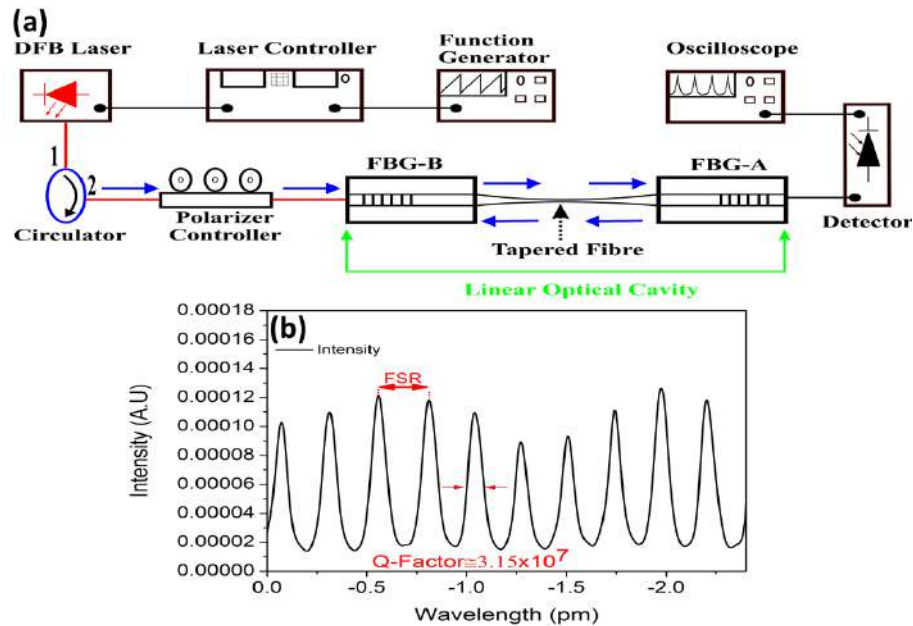


Figure 3.16: (a) The layout of apparatus used for recording the operation of optical cavity, (b) transmission spectrum from cavity observed with the help of photo detector.

Quality factor is the unit-less quantity, that serves as a parameter to indicates the energy losses of the resonator. Lower the losses higher will be the Q-factor and vice versa. For an optical resonator it is given by

$$Q = \frac{\lambda}{\Delta\lambda} = 2\pi \frac{\text{Energy stored}}{\text{Energy lost per cycle}}. \quad (3.9)$$

for our linear optical resonator this value came out to be “ 3.15×10^7 ” for the central wavelength “ λ ” of 1550 nm. Q-factor is related to the ring down time “ τ ” of the cavity by Equation 3.10 as follows.

$$\tau = \frac{Q\lambda}{2\pi c}. \quad (3.10)$$

Where “c” is the speed of light taken to be $3 \times 10^8 \text{ms}^{-1}$. From Equation 3.9 ring down time computed about $25.91 \times 10^{-9} \text{s}$. Intensity of the light “I” exiting the cavity is related to the light entering in it, with the help of Q-factor as

$$I(t) = I_o e^{-t \frac{\omega}{Q}}. \quad (3.11)$$

Detailed derivation of the equation can be found in appendix. So, there is the corresponding change in the output light intensity with the increase in energy lost. This behaviour is quite visible in Figure 4.4 (b). For the purpose of real-time sensing, analyte of interest should affect or bring change in either Q-factor or ring down time without damaging the FBGs or tapered section of the cavity. Tapered optical fiber between the two FBGs acts as sensing probe, that can detects the changes in the surroundings.

Chapter 4

LINEAR CAVITY TAPERED FIBER SENSOR USING MODE-TRACKING PHASE-SHIFT CAVITY RING DOWN SPECTROSCOPY

4.1 Introduction

This chapter presents linear cavity tapered fiber sensing by using mode-tracking phase shift cavity ring down spectroscopy. Cavity ring-down spectroscopy (CRDS) is a spectroscopic technique that is based on measurement of decay rate of light in a high-finesse cavity for detection of minute changes in absorption and scattering losses of the cavity [Wheeler et al., 1998]. In conventional CRDS, photon lifetime (ring down time) is measured via fast switching of an excitation laser and measurement of the photon decay time by an exponential fit of the cavity output. Results are then correlated with the optical losses of the cavity [O’Keefe and Deacon, 1988, Berden and Engeln, 2009]. The CRDS technique has various applications including breath analysis [Wang et al., 2009, Wang and Surampudi, 2008], chemical detection [Wang and Mbi, 2007, Vasudev et al., 1999, Van der Sneppen et al., 2009], biosensing [Tarsa et al., 2004b] and RI sensing [Ni et al., 2008, Sharma et al., 2017]. Experimentally, CRDS setups require the use of pulsed lasers or fast optical modulators together with fast electronics for data acquisition, since the measured ring down times can take values as small as around 10^{-9} s. To extract ring down times, exponential fitting procedures are then required, which may become inconvenient for real-time biological sensing applications [Tong et al., 2004].

To reduce uncertainties in measurements, CRDS systems are also often augmented with the Pound–Drever–Hall (PDH) technique for stabilizing the laser source [Long et al., 2012, Avino et al., 2014, Burkart et al., 2014]. The use of PDH locking adds

more complexity to the overall setup. For a real-time sensing application along with a simpler setup, phase-shift cavity ring down spectroscopy (PS-CRDS) can be utilized. In PS-CRDS, a laser beam is intensity modulated and relayed to the cavity input, and the resultant beam from the cavity output differs in phase and modulation depth compared with the input [Engeln et al., 1996]. The phase measurement then provides necessary information for the calculation of the precise ring down time. Previous PS-CRDS experiments for liquid phase applications employed either fiber loop resonators [Tong et al., 2004] or microresonators such as microspheres [Barnes et al., 2013] or microtoroids [Cheema et al., 2012]. For the demonstrations using fiber loop resonators, narrow-bandwidth CW lasers that were kept at a fixed wavelength were used for excitation of relatively long fiber loop cavities (>20 m cavity length). These demonstrations could not benefit from sweeping of the excitation laser wavelength for coupling to individual cavity modes due to the laser spectral bandwidth being comparable to or larger than the free spectral range (FSR) of the fiber loop resonator. In the demonstration by Tong et al., concentration of 1,1'-diethyl-4,4'-dicarbocyanine iodide was detected in dimethyl sulfoxide with a minimum detection limit $\sim 6 \mu\text{M}$ [Tong et al., 2004].

Detection of small micromolar concentrations in a 100 nL liquid volume was also demonstrated using a scheme combining PS-CRDS with frequency multiplexing at two different wavelengths [Waechter et al., 2011]. On the other hand, PS-CRDS studies using microresonators achieved coupling to individual cavity modes by laser wavelength sweeping thanks to the large FSRs granted by short cavity lengths. This enabled PDH locking of the laser to a cavity mode [Barnes et al., 2013] or simultaneous measurements of the phase shift with the quality factor (Q factor) and resonant wavelength by using wavelength scanning [Cheema et al., 2012]. Here we demonstrate PS-CRDS measurements using a small linear fiber resonator with total length less than ~ 2 m consisting of two fiber Bragg gratings (FBGs) and a tapered optical fiber. When compared with microresonators, our cavity is easy to build and is free from problems associated with evanescent coupling of a tapered fiber to a microsphere or microtoroid [Barnes et al., 2013, Cheema et al., 2012]. At

the same time, the small length of the linear fiber resonator provides a large enough FSR that can readily be resolved in the transmission and phase-shift spectra. Such an analysis, in which coupling to individual cavity modes is allowed, is not possible when relatively long fiber loop resonators are used. Hence, our experimental scheme of using a linear fiber resonator possesses advantages over both fiber loop resonators and microresonators when PS-CRDS is considered. For improvements in the measurement uncertainty, we used a wavelength-scanning approach instead of the PDH locking technique and thus simplified the overall experimental setup. In this wavelength-scanned PS-CRDS approach, we simultaneously employed direct current modulation and external amplitude modulation of the laser as shown in Fig. 1(a). The direct current modulation provides the wavelength scanning, and the external modulation enables the PS-CRDS measurements. The current modulation should be slow enough to provide the necessary buildup time for the cavity. This wavelength-scanned PS-CRDS enabled us to conduct continuous phase-shift measurements at each point of the wavelength-dependent transmission response. Previously, this approach has only been used with microresonators [Cheema et al., 2012] but not with any fiber-based CRDS schemes. We employed a peak tracking algorithm scheme to analyze the resonant phase shifts corresponding to individual cavity modes. Analyzing only the resonant phase shifts, we determine sucrose concentration in deionized (DI) water. Our measurements revealed limit of detection (LOD) values ranging between 121 and 441 μM for sucrose concentration sensing in DI water. When converted to refractive index, these correspond to 121 LOD values between 4.53×10^{-6} - 1.58×10^{-5} RIU.

4.2 Experiment and Data Analysis

Figure 4.1(a) shows the experimental setup used for PS-CRDS measurements. A linear fiber resonator built using two FBGs with $\sim 86\%$ reflectivity, and a tapered optical fiber was used in the experiments. The tapered fiber, which was immersed in a sample chamber, served as the sensing probe. In order to fabricate the tapered fibers, a standard telecom single-mode fiber with a wavelength range of 1260–1650

nm (Thorlabs SMF-28) was stripped of its protective coating and cleaned with isopropyl alcohol. The fiber patch was placed onto the fiber holders mounted on computer-controlled motorized stages. Tapering was performed by pulling the optical fiber with the motorized stages, while simultaneously heating the fiber with the hydrogen flame. During tapering, hydrogen flow rate was maintained at 95 ml/min. Depending on the total elongation distance of two motorized stages (L_{total}), fiber taper diameters (D_{taper}) of 3–6 μm were obtained at the end of the pulling procedure. For sensing purposes, such a taper diameter was sufficient for the efficient interaction of the evanescent field with the surrounding medium.

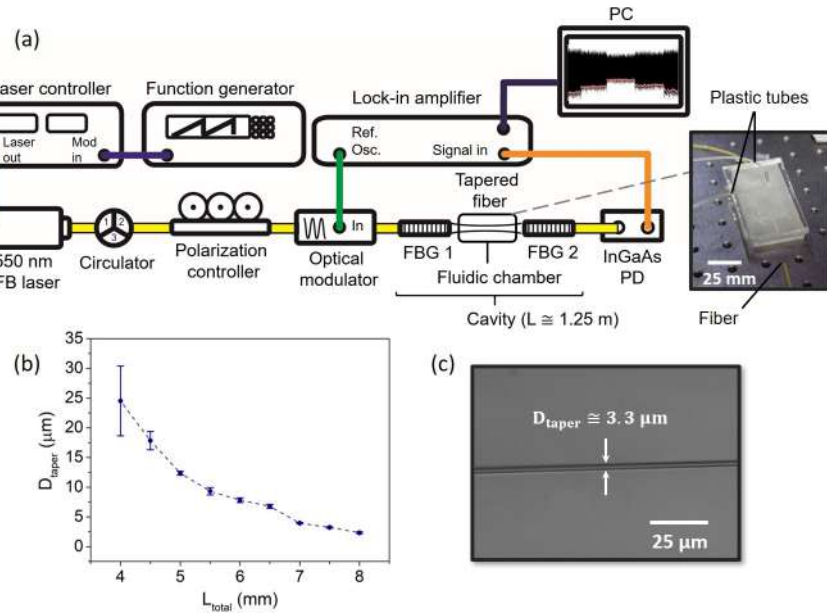


Figure 4.1: (a) Schematic of the experimental setup used for sucrose concentration sensing using a linear cavity tapered fiber sensor. The inset shows a picture of the fluidic chamber used in experiments, (b) dependence of the fiber taper diameter on the total elongation distance of two motorized stages used during fiber tapering by heat and pull method, (c) optical microscope image of a tapered fiber with a diameter of 3.3 μm .

Following the completion of tapering, the hydrogen flame was turned off, and additional pulling of 50 to 70 μm was performed to avoid bends in the tapered region.

The fabricated tapered fiber was fixed on a U-shaped plastic fixture with the help of an epoxy glue. This fixation simplifies handling and ensures steady operation during the injection of fluid into the chamber containing the tapered fiber. The tapered fiber assembly was then rapidly placed into the fluidic chamber to avoid damage and contamination of the tapered region. The dependence of (D_{taper}) on (L_{total}) obtained for our experimental conditions is shown in Figure 4.1(b). This calibration curve was obtained by measurement of (D_{taper}) values through optical imaging immediately after fiber tapering performed with different (L_{total}) values. An exemplary image of a tapered fiber is shown in Figure 4.1(c). Based on optical images and (L_{total}) values of the tapered fibers, we estimate that the typical length and diameter of tapered fibers were less than 5 mm and 6 μm , respectively, in our reported experiments. Following the fixation of the tapered fiber, two single-mode optical fiber pigtail connectors were spliced onto the end faces of the tapered optical fiber using a core alignment fusion splicer (Fujikura 70s). The pigtail connectors were joined with FBGs via FC/APC single-mode simplex adapters and thus formed a cavity. The length of the cavity was 1.25 m.

Finally, a plastic tube was attached on the fluidic chamber for the injection of the test liquids. The fabricated fiber-based optical cavity was connected to a pigtailed distributed feedback (DFB) laser (model 1751-C03 AWDM laser module) centred at 1550 nm. The DFB laser was amplitude modulated with a sinusoidal pattern at a modulation frequency between 1 and 8 MHz via an external electro-optical intensity modulator (Sumitomo Osaka Cement Co. LTD T.MHz 1.5-10PD-ADC). Furthermore, driving current of the DFB laser was modulated using a ramp pattern generated by the function generator (RIGOL DG4202) at 40 mHz, enabling the laser wavelength to be swept within a 10.1 pm wavelength range. As the laser wavelength was swept, the matching cavity modes were excited at resonant wavelengths. The output of the cavity was monitored via an InGaAs photodiode (Thorlabs DET10C/M). The photodiode signal was demodulated with a lock-in amplifier (Zurich Instruments HF2LI) for the extraction of the cavity throughput and phase information. PS-CRDS relies on the phase shift between the laser signal pumped

into the cavity and transmitted out of the cavity being $\phi = -\arctan(\omega \tau)$, where τ is the ring down time and $\omega = 2\pi f$ is the modulation frequency [Tong et al., 2004]. The negative sign of ϕ indicates a lag of the output signal compared to the input signal. The ring down time of the cavity is given as $\tau = Q\lambda_r/2\pi c$, where Q is the Q factor, c is the speed of light, and λ_r is the resonant wavelength ($\lambda \sim 1550$ nm in our case). Hence, any mechanism that introduces absorption or scattering loss in the cavity will affect the Q and τ , and induce a shift in ϕ . This phase shift serves as the sensitive indicator of the changes in the external environment. Measurements were initiated with the injection of DI water into the fluidic chamber in order to establish a baseline measurement of the experimental arrangement. After the DI water, sucrose solutions of various concentrations were introduced into the chamber at regular intervals.

The data collected via the lockin amplifier was later analyzed using a series of programs written in MATLAB. The analysis of the data consists of extraction of the phase values of individual cavity modes. Initially the lock-in data was divided into data sets at the start of each individual wavelength sweep. A typical phase pattern generated during a single laser sweep is shown in Figure.4.2.

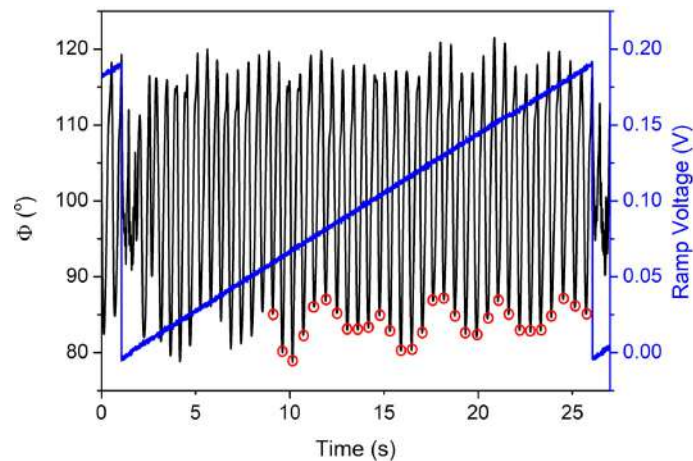


Figure 4.2: Phase values measured during a single exemplary laser sweep, red circles indicate the phase peaks of 30 cavity modes tracked along all wavelength sweeps.

At the beginning of each wavelength sweep, due to the abrupt nature of the transi-

tion of the ramp signal, there exists an unstable region where the laser is settling. In order to exclude this settling period, wavelength sweeps in each dataset were inverted in time. After this inversion, the peak phase values of the first 30 cavity modes were numbered and tracked for all wavelength sweeps. Peak phase and transmission values determined for an exemplary sweep are shown with red circles in Figure 4.2. For each sweep, a single peak phase value ϕ_{peak} and a single peak transmission value T_{peak} were then calculated by averaging of the peak phase values and peak transmission intensities, which were determined for 30 cavity modes. The extracted phase and transmission information contains steps corresponding to different concentrations of the solution under test. In order to convert the measured phase values to refractive indices, an average phase shift for a concentration step is calculated by averaging the ϕ_{peak} values of sweeps at a given concentration step. Using three concentration steps within a measurement and their known refractive indices, a second-order fit between the phase and refractive indices is performed [Saunders et al., 2016].

4.3 Results and Discussions

The results of our PS-CRDS verification experiments are shown in Figure 4.3. These experiments were performed using a tapered fiber with $D_{\text{taper}} = 3.9 \mu\text{m}$ having air as the surrounding medium. In these experiments, peak-to-peak phase-shift values (ϕ_{pp}) that correspond to the average peak-to-peak phase observed during laser sweeps were calculated for different modulation frequencies in the range of 1–8 MHz using air as the sensing medium. The use of ϕ_{pp} eliminates the phase delays caused by the optical and electronic signal transmission and detector response [Barnes et al., 2013]. Figure 4.3 also shows the theoretically expected peak-to-peak phase-shift values calculated using $\phi = -\arctan(\omega\tau)$ based on cavity Q factors measured from laser transmission data. The expected peak-to-peak phase shift values are calculated assuming an average Q factor of 1.79×10^7 , which is the average of measurements performed for eight different frequencies between 1 and 8 MHz. The Q-factor dependence on modulation frequency is also investigated, and significant change is not

observed [Figure 4.3(a), inset]. A good overlap is observed between the measured and calculated ϕ_{pp} values presented in Figure 4.3. Peak-to-peak phase magnitude of 28.5° was observed at 6 MHz modulation frequency.

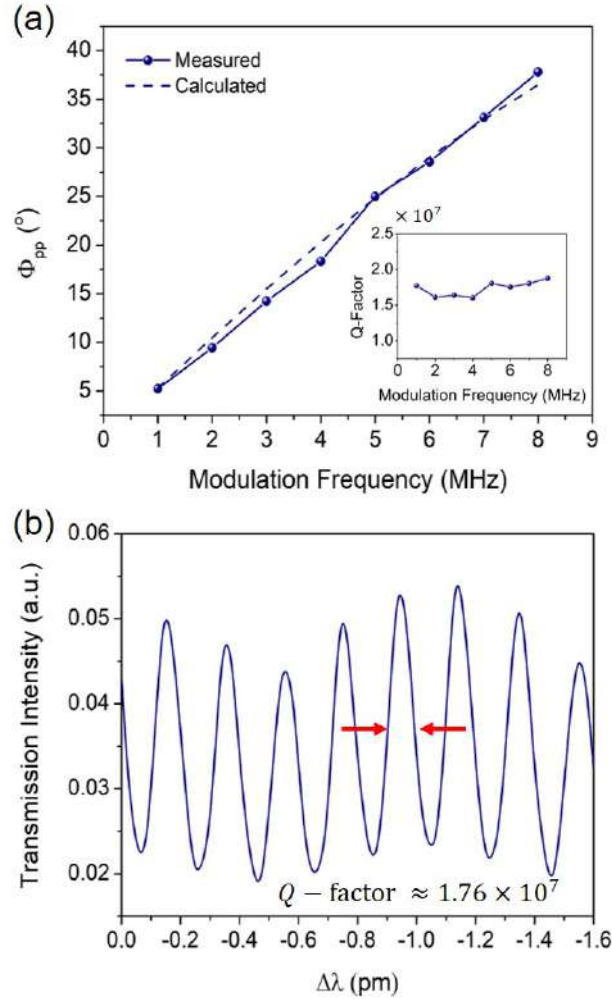


Figure 4.3: (a) Measured (solid line) and calculated (dashed line) peak-to-peak phase-shift values at different modulation frequencies for a linear cavity fiber taper sensor in air. The dashed line is calculated assuming an average Q factor of 1.79×10^7 . The inset shows the dependence of Q factor on the modulation frequency, (b) exemplary transmission spectrum recorded at 6 MHz modulation frequency revealing a Q-factor of around 1.76×10^7 .

In the following step, sensitivity experiments were performed with the sucrose con-

centration increasing and decreasing between 0, 2.9, and 5.8 mM. Figures 4.4(a) and 4.4(b) show the raw data of phase angles ϕ and transmission intensities recorded during such an exemplary experiment together with ϕ_{peak} and T_{peak} values calculated for each ramp sweep. A decrease in peak-to-peak phase angle (ϕ_{pp}) with increasing sucrose concentration due to the rise of cavity losses is clearly visible in this figure, consistent with the verification experiments shown in Figure 4.3.

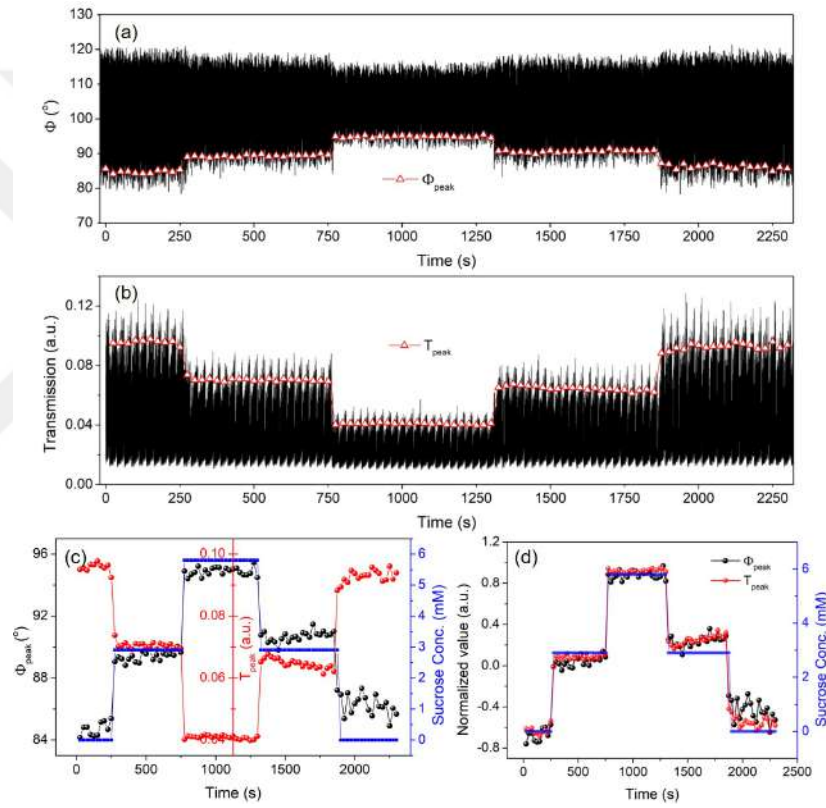


Figure 4.4: Raw data showing the change in (a) phase angle and, (b) transmission intensity measured as the sucrose concentration surrounding the linear cavity tapered fiber sensor was changed. Red symbols indicate the ϕ_{peak} and T_{peak} values calculated for each laser sweep, (c) time traces of ϕ_{peak} and T_{peak} values plotted together with the change in sucrose concentration, (d) correlation plot that shows the time traces of ϕ_{peak} and T_{peak} data sets after proper normalization and offsetting plotted together with the change in sucrose concentration.

Similarly, T_{peak} also decreases with increasing sucrose concentration and cavity loss.

ϕ_{peak} and T_{peak} values calculated for each laser sweep are also shown in Figure 4.4(c) together with traces showing the change in sucrose concentration with time. Figure 4.4(c) shows clear increases in ϕ_{peak} and decreases in T_{peak} with increasing sucrose concentration. In order to compare the behaviour of phase with respect to transmission, a plot showing the correlation between the phase and transmission data was generated [Figure 4.4(d)]. In order to do this, both transmission and phase data were centred to zero by subtracting their mean values and normalized by dividing both data sets by their absolute maximum values. The transmission data shown in this plot is inverted since the transmission and phase are inversely proportional. Figure 4.4(d) shows very good correlation between two data sets. Drifts in phase and transmission were observed within certain time intervals, and an overall offset was observed between initial and final values of ϕ_{peak} and T_{peak} time traces. These are attributed to the changes in the overall cavity transmission that are mainly caused by the instabilities due to the tapered fiber that may be caused by thermal fluctuations or the deposition of contaminants on the tapered fiber surface. Fluctuations in the time traces of ϕ_{peak} were analyzed in order to obtain the LOD in sucrose concentration measurements. At each sucrose concentration step shown in Figure 4.4(c), we calculated the standard deviation of ϕ_{peak} data and converted the resulting sensitivities to sucrose concentrations and to refractive index changes. For consecutive concentration steps in Figure 4.4(c), LOD values were calculated as 441, 181, 121, 192, and 394 μM . When converted to refractive index, these values correspond to LOD values of 1.25×10^{-5} , 6.70×10^{-6} , 4.53×10^{-6} , 7.22×10^{-6} , and 1.58×10^{-5} RIU, respectively [Saunders et al., 2016]. We note that when the above analysis was performed for T_{peak} time traces, comparable LOD values were obtained with slight improvements by up to a factor of 2.

These LOD values obtained by ϕ_{peak} measurements constitute an improvement over the LOD value of 1.03×10^{-4} RIU reported in sucrose sensing experiments using conventional, amplified, and gain clamped CRDS measurements [Sharma et al., 2018, Liang et al., 2005]. This improvement is mainly attributed to: (i) the relatively high modulation frequency of 6 MHz together with lock-in demodulation

used in our measurements and (ii) improvements in eliminating non-resonant phase fluctuations by only considering the peak phase values of individual cavity modes via the mode-tracking approach.

The diameter of the fiber taper (D_{taper}) has a direct impact on the sensitivity of our sucrose concentration sensor. Figure 4.5 shows the average ϕ_{peak} values measured as a function of sucrose concentration using tapered fibers with D_{taper} values changing between 3.2 and 5.4 μm , whereas the sucrose concentration was first increased from 0 to 5.8 mM (solid lines) and then decreased back to 0 (dashed lines). The inset in Figure 4.5 shows the average phase-shift sensitivity expressed in units of $^{\circ}/1 \text{ mM Suc.}$ as a function of D_{taper} observed in experiments performed with different fiber tapers.

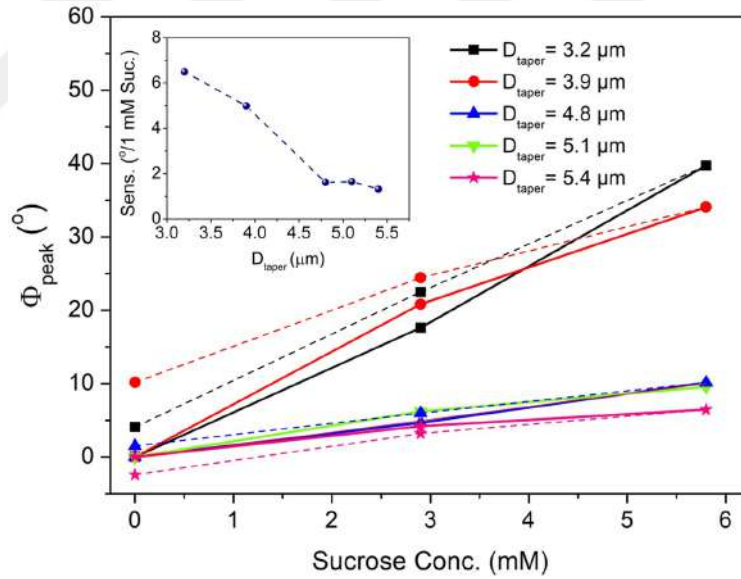


Figure 4.5: Sucrose concentration dependence of average ϕ_{peak} values for increasing (solid lines) and decreasing (dashed lines) directions for five different experiments with different D_{taper} values. The inset shows the dependence of the sensitivity of the linear cavity tapered fiber sucrose concentration sensor on D_{taper} .

For this plot, the sensitivity was calculated as the average of the sensitivities observed during increasing and decreasing directions of Figure 5.5. The sensitivity of ϕ_{peak} on sucrose concentration was observed to increase from 1.3 to 6.5 $^{\circ}/1\text{mM}$

Suc. when D_{taper} was decreased from 5.4 to 3.2 μm . This change in sensitivity is consistent with the increasing optical coupling between the liquid mixture and tapered fiber with decreasing D_{taper} . For D_{taper} values smaller than around 3 μm , cavity transmission could not be detected, since the cavity losses were dominated by the absorption of water. For D_{taper} values smaller than around 3 μm , cavity transmission could not be detected, since the cavity losses were dominated by the absorption of water. Hence, in our experimental conditions 3–5 μm was determined to be the optimum D_{taper} range, allowing maximal sensitivity and manageable levels of water-absorption-induced cavity losses. In the future, sensitivity can further be increased by addition of a fiber amplifier into the fiber cavity in order to compensate for the cavity loss due to water absorption at smaller fiber taper diameters.

In order to show the repeatability of the sensing performance, cyclic injections of sucrose solutions with concentrations varying between 0, 2.9, and 5.8 mM were performed. Figure 4.6 shows the time trace of the ϕ_{peak} values observed during four such cycles, in which good repeatability was observed.

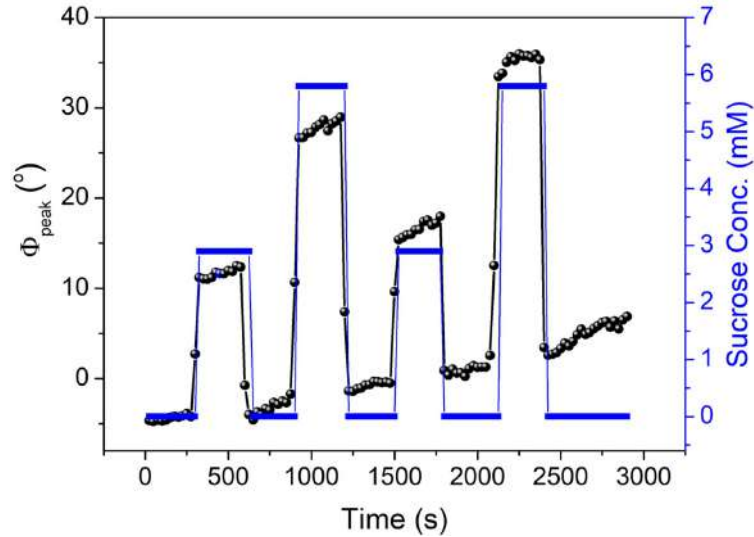


Figure 4.6: Time trace of peak phase shifts recorded during a cyclic experiment in which sucrose concentration changed between 0, 2.9, 0, and 5.8 mM, consecutively. The black curves denote the measured phase shifts; the blue lines denote the actual sucrose concentrations.

A relatively fast response with response time smaller than around 20 s was observed at each cycle. Good sensitivity of around $5.3^\circ/1 \text{ mM}$ was observed during each cycle. The constant drift observed in ϕ_{peak} agrees with the change in transmission intensity, consistent with the observations made in Figure 4.4(d).



Chapter 5

AMPLIFIED LINEAR CAVITY TAPERED FIBER SENSOR USING AVERAGE PHASE SHIFT CAVITY RING DOWN SPECTROSCOPY

5.1 Introduction

Phase shift cavity ring down spectroscopy is a popular and quite mature sensing method [Cheema et al., 2012, Tong et al., 2004, Reid et al., 2011]. PS-CRDS not only has the advantages of classical CRDS techniques but also cover up its limitations. PS-CRDS involves the measurement of change in phase shift as a function of cavity losses and use it as its sensing mechanism. For this reason it does not need any costly electronic systems without compromising on its working efficiency. In previous chapter, we reported the linear cavity tapered fiber based sensor which is capable of measuring the concentration change in the range of 121 and 441 μM [Ayaz et al., 2020] with corresponding 4.53×10^{-6} - 1.58×10^{-5} in terms of RIUs.

The sensitivity of the reported sensor shows a relation to the taper waist diameter. That is, thinner the waist then higher will be the sensitivity and vice versa. But it is unfortunate fact that, in addition to most of organic molecules, water also has strong absorption at 1550nm [Walsh, 2009] and serve as a lossy medium. This phenomenon severely affects the sensitivity and limit of detection of all those liquid based absorption measurements methods that make use of water to establish base line. In addition to water absorption, there are many other sources of losses. Optical losses can be due to use of optical consumables for making connections, fiber splicing, any kind of irregularity (e.g., unobservable sag) in taper region while glueing or placing tapered fiber containing PCB mould into fluidic chamber. The sum of all these said loss mechanisms results into lesser photon life time, short effective absorption path

lengths and noisy output signal with less intensity. For the purpose of improving the limit of detection and sensitivity of the device by overcoming or compensating for the losses, there was the dire need for alternative arrangement. A reasonable answer to this problem is the incorporation of optical signal amplifier into the optical sensing setup. Signal amplification can be achieved by using either of an erbium doped fiber amplifier (EDFA) or booster optical amplifier (BOA). The use of optical signal amplifiers for increasing sensor performance by dominating over the cavity losses is not a new concept. There are numerous studies available in the literature that describe the use of optical signal amplifiers along-with standard CRDS for such purposes. These include the use of EDFA in fiber loop for the purpose of refractive index sensing [Silva and Frazão, 2017] giving sensitivity of $580 \mu\text{s}/\text{RIU}$ [Silva and Frazão, 2017] and limit of detection about $7.8 \times 10^{-5} \text{ RIU}$ [Wong et al., 2012]. In another case that involves the gas detection [Atherton et al., 2002], increase of ring down time changed effective path length from 5cm to 17m. Determination of the cavity loss [Stewart et al., 2001] is one more classical example for the use of signal amplifier. In addition there is a investigation that compares the performance of optical amplifier based CRDS system to normal CRDS once and hold former superior in terms of limit of detection [Sharma et al., 2018]. Since amplified CRDS techniques is similar in all aspects to standard CRDS except it uses optical amplifier. So, it inherits the same limitations that has been described in chapter 2 and 4.

Application of optical signal amplifier on PS-CRDS method in linear cavity tapered fiber arrangement and its possible effect on the working of the PS-CRDS based device is topic, that has not been investigated so far. This chapter explores the possibility of improvements that can be realized by using amplified version of simple PS-CRDS. It is known that ring down time “ τ ” of the cavity [Brown et al., 2002] is given by

$$\tau = \frac{\text{Round trip time}}{\text{Sum of cavity losses}}. \quad (5.1)$$

Cavity losses can be further categorised into two kinds. First kind belongs to those losses which are intrinsic in nature. These include losses at spliced points, optical connectors, taper optical fiber and due to liquid containing analyte of interest (for

liquid phase analysis). Those loss that are caused because of the presence of analyte to be studied fall in to the second kind. But with the addition of optical amplifier Equation 5.2 can be modified as

$$\tau = \frac{\text{Round trip time}}{\text{Sum of cavity losses} - G}. \quad (5.2)$$

In Equation 5.2, “G” represents the gain of the amplifier used to compensate for losses of the cavity. For increasing the ring down time, gain is varied in systematic manner to avoid lasing [Silva et al., 2016]. The gain is varied to point which ultimately leaves only those losses which are caused by the analyte of interest. Since ring down time is related to the phase shift by $\tan\phi = -\omega\tau$, in this way sensitivity and LOD can be increased.

5.2 Experiment and Data Analysis

Experimental arrangement for the amplified PS-CRDS has been shown in Figure 5.1. For the experiment a loss compensated linear resonator was deployed. It comprised of two ready made FBGs with reflectivity $\sim 86\%$, 1×2 coupler, booster optical amplifier, circulator and tapered optical fiber. Sensing region composed of tapered fiber, was dipped into the sample containing fluidic chamber. This sample container used to have proper openings for the injection and removal of the fluids. For the manufacturing of the sensing element, regular telecom SMF was converted to final tapered geometry. Here fiber tapering was done, until fiber taper diameter (D_{taper}) of about $\sim 1.75 \mu\text{m}$ or less was achieved. For having optimum interaction between evanescent field and surrounding environment for the purpose of maximum sensitivity, this taper diameter was adequate. This tapered structure spliced with two pigtailed, one on each side. This whole process that results into taper assembly containing two pigtailed involves sequence of steps. This sequence begins with the fiber stripping, then cleaning with IPA, followed by heating with pulling and at the end splicing with pigtailed by using fusion splicer. The details of these steps has been given in chapter 4. Compared to the simple PS-CRDS, in amplified PS-CRDS light coming from the first FBG was split into two equal parts having same intensity with

The diagram illustrates the experimental setup for the optical cavity. The components and their connections are as follows:

- DFB Laser**: The light source, connected to the **Laser Controller** and **Function Generator**.
- Laser Controller**: Controls the laser's output, connected to the **Function Generator** and the **Intensity Modulator**.
- Function Generator**: Provides a reference signal to the **Lockin Amplifier** and the **Intensity Modulator**.
- Computer**: Controls the **Lockin Amplifier** and the **Intensity Modulator**.
- Lockin Amplifier**: Processes the signals from the **Detector** and the **Function Generator**.
- Detector**: Detects the light signal from the **Circulator**.
- Isolator**: Prevents back-reflection from the **Polarizer Controller** to the **DFB Laser**.
- Polarizer Controller**: Controls the polarization of the light, connected to the **Intensity Modulator**.
- Intensity Modulator**: Modulates the light intensity, connected to the **FBG-A** and the **Tapered Fiber in Fluidic Chamber**.
- FBG-A**: Fiber Bragg Grating, connected to the **Intensity Modulator** and the **1X2 Coupler**.
- Tapered Fiber in Fluidic Chamber**: The fiber under investigation, connected to the **Intensity Modulator** and the **1X2 Coupler**.
- BOA**: Bandwidth Optimized Amplifier, connected to the **1X2 Coupler** and the **Circulator**.
- 1X2 Coupler**: Splits the light signal into two paths, connected to the **FBG-A** and the **Circulator**.
- Circulator**: Directs the light signal to the **FBG-B** and the **Detector**.
- FBG-B**: Fiber Bragg Grating, connected to the **Circulator** and the **Detector**.
- Optical Cavity**: The overall system, including the **DFB Laser**, **Isolator**, **Polarizer Controller**, **Intensity Modulator**, **FBG-A**, **Tapered Fiber in Fluidic Chamber**, **BOA**, **1X2 Coupler**, **Circulator**, **FBG-B**, and **Detector**.

In the last step, plastic tubing for exchange of test liquids with the fluidic chambers were attached. A pigtailed distributed feedback (DFB) laser (model 1751-C03 AWDM laser module) centered at 1550 nm was connected to fibrous optical cavity. An external electro-optical intensity modulator (Sumitomo Osaka Cement Co. LTD T.MHz 1.5-10PD-ADC) was used to achieve amplitude modulation of DFB laser at 2 MHz. Moreover, DFB laser was kept within 10.1 pm wavelength sweep range by modulating its driving current at the frequency of 5 Hz in the form of ramp

pattern generated by function generator (RIGOL DG4202). Sweeping of laser wavelength resulted into matching cavity modes at resonant wavelengths. An InGaAs photodiode (Thorlabs DET10C/M) was used to observe the cavity output. Typical transmission pattern coming from loss compensated linear cavity is shown in Figure 5.2.

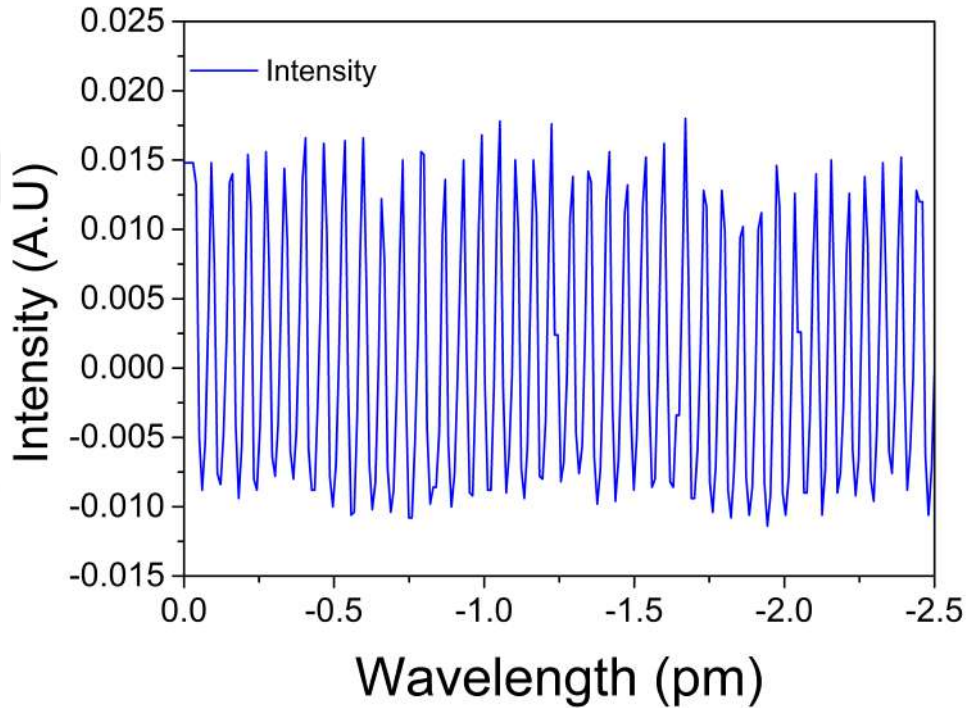


Figure 5.2: An exemplary transmission spectrum recorded at the gain of 120 mA and 5 Hz scanning frequency.

Because of additional optical components between two FBGs, the length of cavity increased. Then correspondingly more dense mode pattern was observed. For the extraction of phase information, signal from photodiode (Thorlabs DET10C/M) was demodulated using lock-in amplifier (Zurich Instruments HF2LI).

Just like simple PS-CRDS, its variation amplified PS-CRDS depends on phase shift that exists between the input and output of the cavity, related to ring down time as $\phi = -\arctan(\omega\tau)$, where τ is ring down time and $\omega = 2\pi f$ is angular modulation frequency [Tong et al., 2004]. The negative sign of ϕ symbolizes that transmitted

signal lags the input signal. The relationship among the resonant λ_r wavelength ring down time τ and quality factor Q of cavity is given as $\tau = Q\lambda_r/2\pi c$. Where c is the speed of light. So, any analyte directly or indirectly enhancing cavity losses via absorption, scattering or both, will affect the Q and τ and ultimately shift to ϕ . This change in phase serves as sensitive parameter to monitor variations in external environment. Data recording were started with the introduction of water in to the fluidic chamber. Compensation of all the losses including absorptive losses of water at 1550 nm were done by suitable adjusting the gain of BOA. The point at which gain of BOA nullified the absorptive loss of water and other associated cavity losses, e.g., caused by splicing, connectors was used as baseline measurement for the experimental configuration. After the DI water, sucrose solutions of different concentrations were administered into the fluidic chamber at fixed time periods. For the injection of each sucrose concentration phase behaviour was observed with the help of lock-in amplifier and data from it was processed further .

The technique that has been used for processing the data of amplified phase shift cavity ring down spectroscopy (APS-CRDS) scenario is different from the simple phase shift cavity ring down spectroscopy. It is because of the fact that in later case, length of the cavity was small (~ 1.25 m). This small length of the cavity made it easier to observe and track the individual cavity mode attitude as a function of concentration change. But in the APS-CRDS length of the cavity is much longer (~ 7 m). Because of length increase and decrease in FSR, observation and tracking of individual cavity mode although not impossible but a difficult job and even in some cases unreliable. To deal with this situation another method was used for analysing the lock-in data. In this scheme, with the help of MATLAB based functions average peak-to-peak phase " ϕ_{pp} " of the modes was calculated, for each session of concentration change. It is done by first dividing the data from lock-in amplifier into packets based on scan frequency of the light source. After that an envelope function is defined for each packet that calculate the average peak-to-peak phase " ϕ_{pp} " difference for each packet.

5.3 Results and Discussions

Gain is the figure of merit for any optical amplifier and it shows its ability to amplify a given weak optical input signal. For example an optical amplifier with a gain of 10 dB can amplify the input by the factor of 10, but gain of 30 dB means an amplification by the factor of 1000 can be achieved. The experimental investigation started with the gain characterization of our booster optical amplifier (BOA) as the function of injection current. For this purpose, output from the laser diode was first subjected to the variable optical attenuator (VOA) to reduce its power to $14.69 \mu\text{W}$ (-18.33 dBm). The basic purpose of this step was to have good gain, lesser noise figure, avoid the saturation of the detector and its damage [Zoiros and Morel, 2014, Wei et al., 2009, Rocha et al., 2018]. On its way after the variable optical attenuator, laser passed through the optical isolator to prevent back reflection of light and ultimately detected by the power meter or optical spectrum analyser (OSA). This measured power was marked as “ P_{in} ” and served the purpose of weak test signal applied at the input port of the booster optical amplifier. The diagrammatic representation for the measurement of “ P_{in} ” is shown in Figure 5.3.

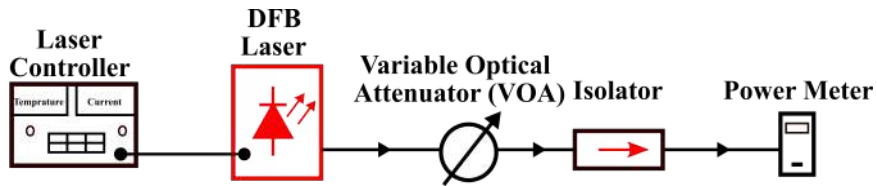


Figure 5.3: Measurement of laser diode power applied at input port of booster optical amplifier.

As last step for gain calculation, current of the booster optical amplifier is varied in equal intervals and corresponding output power “ P_{out} ” is measured. Then by using any of Equations 5.1 or 5.2 gain was calculated and plotted as a function of current

$$\text{Gain(dB)} = 10 \log \frac{P_{\text{out}}(\text{mW})}{P_{\text{in}}(\text{mW})}. \quad (5.3)$$

$$\text{Gain(dB)} = P_{\text{out}}(\text{dBm}) - P_{\text{in}}(\text{dBm}). \quad (5.4)$$

Complete arrangement for measuring the “ P_{out} ” for different values of injection current is depicted in Figure 5.4.

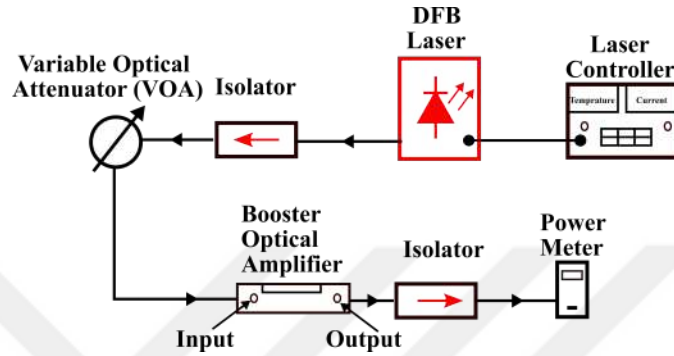


Figure 5.4: Measurement of output power of booster optical amplifier .

The corresponding graph between gain(dB) and current(mA) looks like Figure 5.5. It is quite apparent from the graph that gain increased with the increase of current upto a certain extent. After that, gain becomes almost constant and further current increase can't bring significant change into the gain of the amplifier. It is because of gain saturation of optical amplifier.

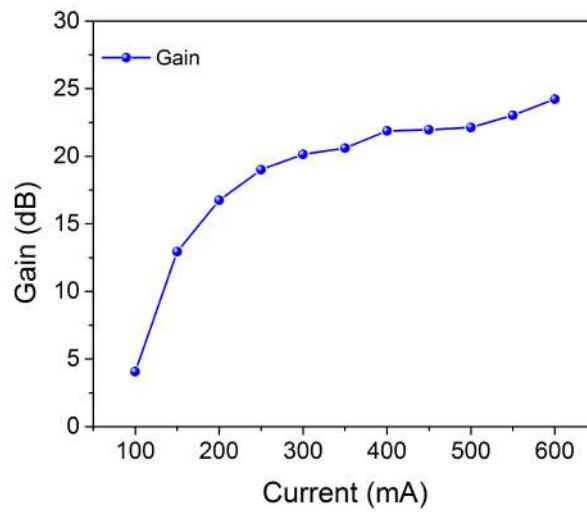


Figure 5.5: Variation of gain with the injection current for booster optical amplifier

The experimental validation results for our amplified PS-CRDS has been shown in Figure 5.6. Behaviour of the system was monitored in air and subsequently in water, with tapered fiber diameter $D_{\text{Taper}} = 1.7 \mu\text{m}$. The gain of booster optical amplifier (BOA) was varied between 100-170 mA. But modulation frequency of the input light signal was maintained constant, i.e., 2 MHz.

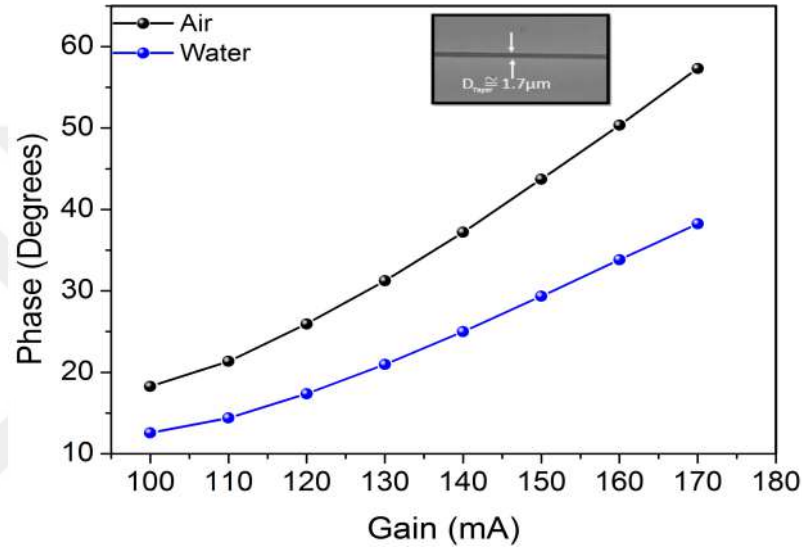


Figure 5.6: Experimental verification of amplified PS-CRDS and inset shows tapered fiber .

From Figure 5.3. it is visible that change in average peak-to-peak phase “ ϕ_{pp} ” increases with the increase of booster amplifier gain in milli amperes (mA). Which in turn enhances the cavity ring down time. This exponential change in “ ϕ_{pp} ” is also in accordance with power current properties of BOA [Cheema et al., 2020]. But this change in average peak-to-peak phase “ ϕ_{pp} ” is different for air and water. It is because of lossy and absorptive nature of water at $\lambda_r = 1550 \text{ nm}$. That is why “ ϕ_{pp} ” has lower values for water compared to air.

After the first step, next objective was to observe the change in “ ϕ_{pp} ” with the change of sucrose concentration to see for the sensitivity of the system. For this purpose, as in previous case, again sucrose solution of 0, 2.9 mM and 5.8 mM concentrations were used as the sample liquids and injected into the fluidic chamber

at regular intervals. In one of the preliminary trials, thicker taper with $\sim 2.2 \mu\text{m}$ waist diameter was used at the gain of about 130 mA. The change in concentration of the sucrose solution affects the phase and transmission that can be seen Figure 5.7.

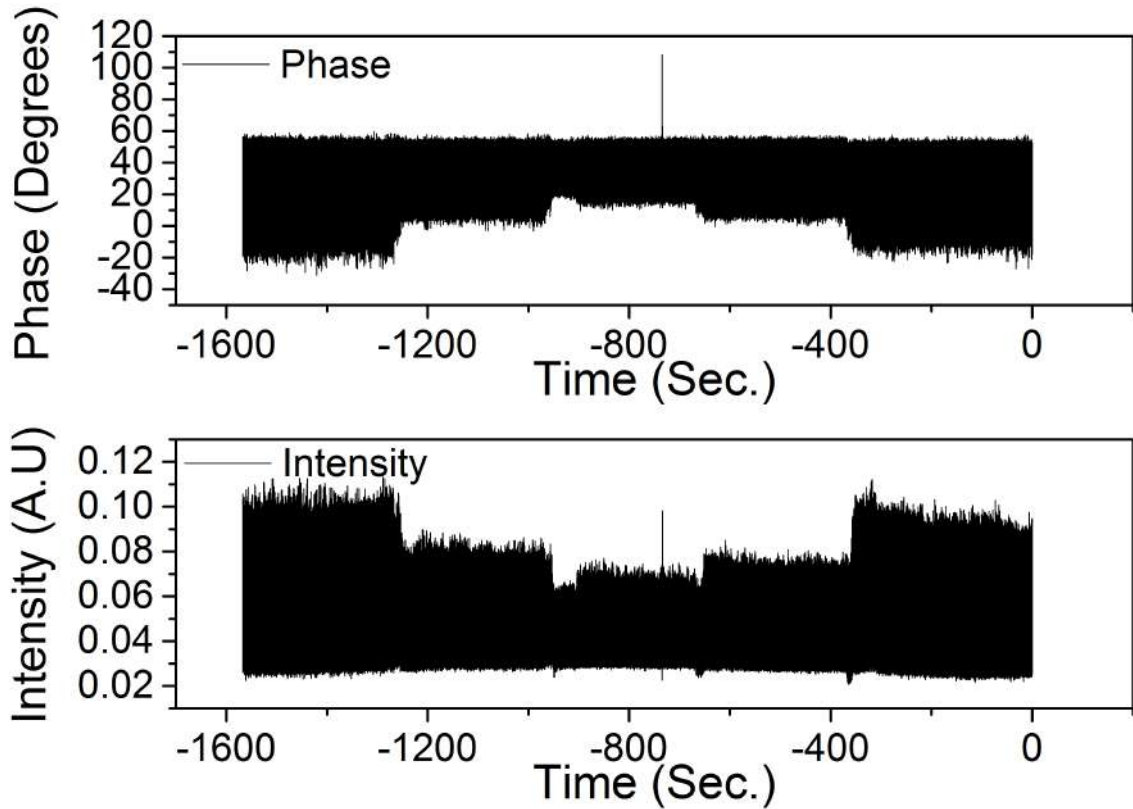


Figure 5.7: Raw data depicting the change in a) phase angle b) transmission with the variation in the sucrose concentration around the tapered optical fiber at the gain of 130 mA.

In the second trial, all the experimental arrangements were kept constant Except the gain of the booster optical amplifier. For the same taper diameter in the second trial gain reduced to 120 mA. The resultant phase and transmission with the change of concentration of sucrose solution can be seen in Figure 5.8. Just like the previous case, change in phase and transmission happens because, change of sucrose solution concentration. This change of solutions concentration as a consequence changes the losses of the cavity. As a result cavity becomes more lossy and in turn affect the

transmission and phase.

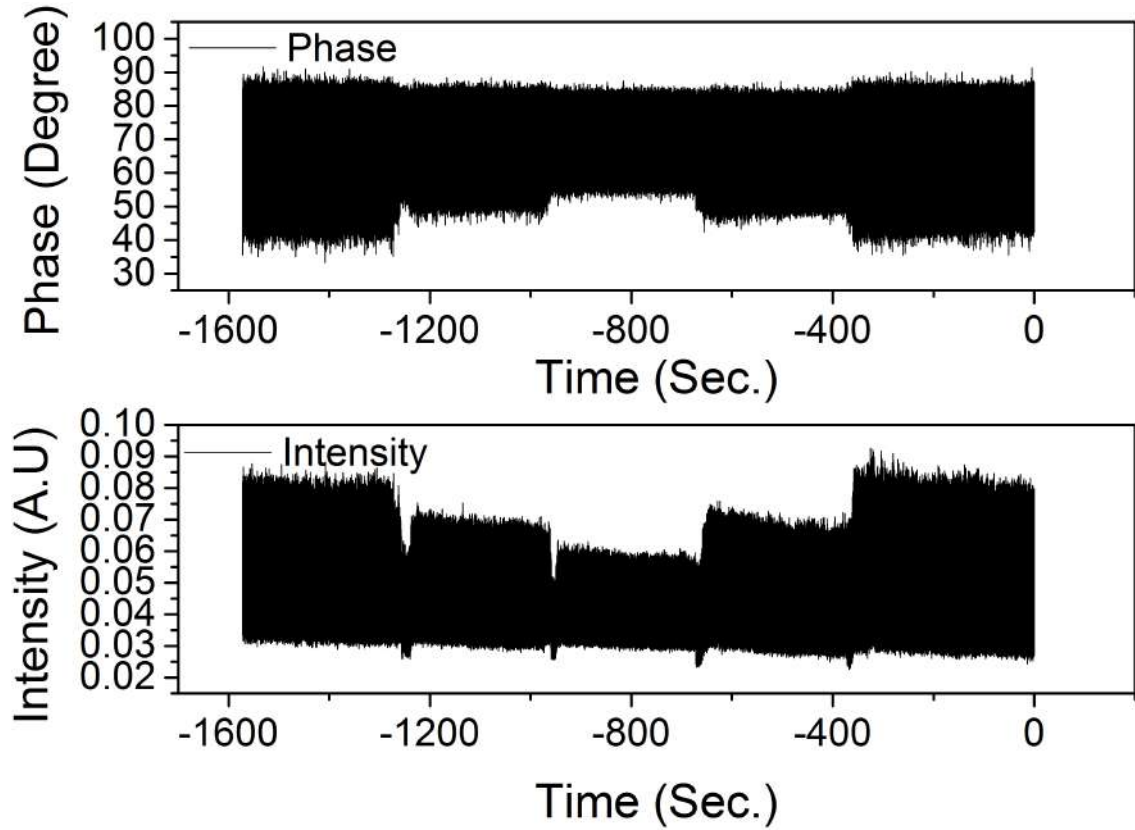


Figure 5.8: Raw data showing the change in (a) phase angle, (b) transmission with the change in the sucrose concentration in the surroundings of tapered optical fiber at the gain of 120 mA.

The observed sensitivity for both of the cases can be seen in the Figure 5.9. When the cavity is maintained at the gain of 130 mA (black line) its sensitivity comes out to be $4.84^\circ/\text{mM}$ giving the limit of detection in the range of 1.19 to 4.39×10^{-5} RIUs. On the other hand, for the gain of about 120 mA (red line) sensitivity calculated to be $2.2^\circ/\text{mM}$, resulting the sensitivity from 2.99 to 7.22×10^{-5} RIUs.

In both of these preliminary trials all the parameters kept constant including the taper waist of the cavity. Only thing that was different in both of these cases was the gain of the booster optical amplifier. The reason behind the difference in sensitivity can be attributed to the loss compensation in both cases. Applied gain of 130 mA

was enough to over come the most of losses of the cavity including water absorption losses. Which means enhanced ring down time and increased sensitivity. But the gain of 120 mA, was not enough to overcome the losses. That affected the ring down time and hence reduced sensitivity was recorded. Amplifier based phase shift cavity ring down systems can be used for the applications where the host liquid absorbs or cause more losses than that analyte of interest.

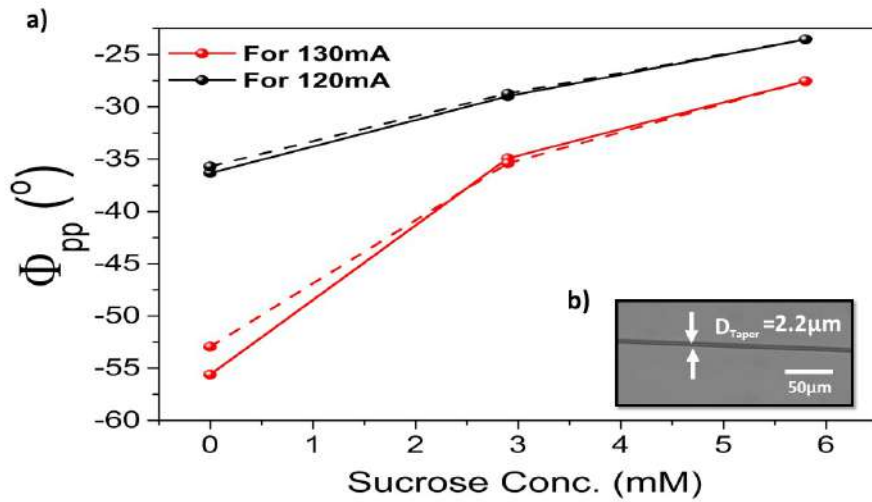


Figure 5.9: (a) Variation in the phase with the increase (solid lines) and decrease (dotted lines) of sucrose solution concentration for two different gains 130 mA (black) and 120 mA (red), (b) inset shows the microscopic image of taper fiber used

Sensitivity can be enhanced even further by reducing the diameter of the taper to even less than $2 \mu\text{m}$ and increasing the gain of amplifier. Provided that the noise associated with optical amplifier is kept constant.

Chapter 6

CONCLUSION AND OUTLOOK

Optical detection and sensing by using linear cavity tapered fiber has been explained in this thesis. Details of principles operating behind making the measurements and techniques for data processing has also been explained. In chapter 1, the needs for the sensing and reason behind having optical sensory systems are described. Then it developed the foundation about the contemporary optical devices available for the purpose of sensing. Their brief comparative analysis in terms of quality factor, system complexity, limit of detection and their constraints are also included in this chapter. This chapter ended with the idea of linear optical resonator and analysis technique for making observations. Spectroscopy and concept behind it are given in chapter 2. Simple direct absorption method, its applications and possible limiting factors in its performance are also included in the same chapter. After simple direct absorption method, discussion on more advanced absorption based techniques like cavity ring down spectroscopy (CRDS) and its variants, e.g., cavity enhanced absorption spectroscopy (CEAS) also belong to same chapter. After maturing the notion about the absorption spectroscopy and its various forms, phase shift cavity ring down spectroscopy (PS-CRDS) is elaborated. Both theoretical and mathematical basis of phase shift cavity ring down spectroscopy are considered. In addition to phase, the possibility of using other parameters like detecting the change in modulation depth with the aim of sensing is also given in chapter 2.

The fundamentals of the components used in the presented thesis for making optical investigations, their fabrication, characterization, principles operating behind them are the part and parcel of chapter 3. Moreover, in chapter 3, the relation between Bragg mirror and Bragg reflector including the mathematical equations that govern their behaviour is explored as well. Information about types of the optical fibers for

example single/multi mode fibers and role of evanescent field for the specific job is in the same section. Optical fiber tapering and its subsequent use for the construction of the linear cavity mark the end of the chapter 3. A highly sensitive FBG based sensor is demonstrated using PS-CRDS with a linear fiber cavity for sucrose concentration sensing in chapter 4. A tapered fiber placed between two FBGs was used as the sensing unit, providing an overall small cavity length that enables tracking of individual cavity modes as a result of laser sweeps.

The mode-tracking approach enabled the observation of relatively small LOD values ranging between 121 and 441 μM which corresponds to 4.53×10^{-6} - 1.58×10^{-5} RIU when converted to refractive index. These LOD values constitute an improvement over LOD value of 1.03×10^{-4} RIU reported in sucrose sensing experiments using conventional, amplified, and gain-clamped CRDS measurements. It was shown that the sensitivity of the sensor highly depends on the diameter of the tapered fiber in the linear cavity tapered fiber sensor. In our experimental configuration, an optimum range of 3–5 μm for taper diameter was obtained as a good compromise between the dominance of water absorption and good sensitivity, and experiments with the tapered fiber that had 3.2 μm diameter revealed the maximum sensitivity around 6° for 1 mM sucrose concentration in water. Through the implementation of amplification technology for enhancement of the sensitivity of the system, it is possible to use thinner tapered fibers in the cavity, allowing highly sensitive detection of small amounts of compounds in liquid medium. In addition, the Q-factor of the cavity can be further improved with higher reflectivity FBGs. In chapter 5, advanced version of PS-CRDS is introduced that make use of optical signal amplifier to compensate for the losses caused by water absorption or fiber consumables. This new experimental configuration at fixed modulation frequency provide the change in phase shift if the gain of the amplifier is varied. But the increase of phase with increase of gain is unequal for different materials. It is because of same reason that is valid for the simple PS-CRDS.

Motivated by the findings, there are some upgrades that can be applied to make the proposed optical detection systems more sensitive, robust and economical af-

fordable. Some them are, use of light emitting diodes (LEDs) as an alternative of laser sources and operatable on portable power supply. Instead of using tapered fiber, sensing element can also be designed by using hybrid fiber for example single mode and multi mode fibers are spliced in way MMF-SMF-MMF to serve as the sensing probe. Other variations can be the use splicing of coreless fiber between two standard optical fibers. Optical fibers spliced with offset can also serve as a tool for interaction with surrounding. The demonstrated configurations also has a good potential to be employed as a tool for sensitive probing of toxic compounds such as aflatoxin or biosensing with a high specificity following proper surface functionalization of the sensing element.

Appendix A

CHAPTER 2

Consider $W = \tan \theta$

then

$$\sin y \pm w \cos y = \sin y \pm \tan \theta \cos y. \quad (\text{A.1})$$

$$= \frac{1}{\cos \theta} (\sin y \cos \theta \pm \cos y \sin \theta). \quad (\text{A.2})$$

$$= \frac{1}{\cos \theta} \sin (y \pm \theta). \quad (\text{A.3})$$

$$\frac{1}{\cos \theta} = \sqrt{1 + \tan^2(\theta)} = \sqrt{1 + w^2}. \quad (\text{A.4})$$

so

$$\sin y \pm w \cos y = \sqrt{1 + w^2} \sin (y \pm \tan^{-1} w). \quad (\text{A.5})$$

Appendix B

CHAPTER 3

$$Q = \frac{\lambda}{\Delta\lambda} = 2\pi \frac{\text{Energy stored}}{\text{Energy lost per cycle}}. \quad (\text{B.1})$$

if photons represents the energy the

$$Q = 2\pi \frac{h\nu I}{h\nu \left(-\frac{dI}{dt}\right) \frac{1}{\nu}} = -\frac{2\pi\nu I}{\left(\frac{dI}{dt}\right)}. \quad (\text{B.2})$$

then

$$\frac{dI}{dt} = -I \frac{\omega}{Q}. \quad (\text{B.3})$$

that can be simplified to

$$I(t) = I_o e^{-t \frac{\omega}{Q}} = I_o e^{-\frac{t}{\tau_c}} \quad (\text{B.4})$$

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