Polymer Microresonator Sensors Embedded in Digital Electrowetting on Dielectric Microfluidics Systems

by

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Dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the Department of Electrical and Computer Engineering in the Graduate School of Duke University

2012
ABSTRACT

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Abstract

Integrated sensing systems are designed to address a variety of problems, including clinical diagnosis, water quality testing, and air quality testing. The growing prevalence of tropical diseases in the developing world, such as malaria, trypanosomiasis (sleeping sickness), and tuberculosis, provides a clear and present impetus for portable, low cost diagnostics both to improve treatment outcomes and to prevent the development of drug resistance in a population. The increasing scarcity of available clean, fresh water, especially noticeable in the developing world, also presents a motivation for low-cost water quality diagnostic tools to prevent exposure of people to contaminated water supplies and to monitor those water supplies to effectively mitigate their contamination. In the developed world, the impact of second-hand cigarette smoke is receiving increased attention, and measuring its effects on public health have become a priority. The ‘point-of-need’ technologies required to address these sensing problems cannot achieve a widespread and effective level of use unless low-cost, small form-factor, portable sensing devices can be realized. Optical sensors based on low cost polymer materials have the potential to address the aforementioned ‘point-of-need’ sensing problems by leveraging low-cost materials and fabrication processes. For portable clinical diagnostics and water quality testing in particular, on-chip sample preparation is a necessity. Electrowetting-on-dielectric (EWD) technology is an enabling
technology for chip-scale sample preparation, due to its very low power consumption compared to other microfluidics technologies and the ability to move fluids without bulky external pumps. Potentially, these technologies could be combined into a cell phone sized portable sensing device.

Towards the goal of developing a portable diagnostic device using EWD microfluidics with an embedded polymer microresonator sensor, this thesis describes a viable fabrication process for the system and explores the design trade-offs of such a system. The main design challenges for this system are optimization of the sensor’s limit-of-detection, minimization of the insertion loss of the optical system, and maintaining the ability to actuate droplets onto and off of the sensor embedded in the microfluidic system. The polymer microresonator sensor was designed to optimize the limit-of-detection (LOD) using SU-8 polymer as the bus waveguide and microresonator material and SiO₂ as the substrate cladding material. The fabrication process and methodology were explored with test devices using a tunable laser system working around a wavelength of 1550 nm using glucose solutions as a refractive index standard. This sensor design was then utilized to embed the sensor and bus waveguides into an EWD top plate in order to minimize the impact of the sensor integration on microfluidic operations. Finally, the performance of the embedded sensor embedded was evaluated in the same manner and compared to the performance of the sensor without the microfluidic system.
The primary result of this research was the successful demonstration of a high performance polymer microresonator sensor embedded in the top plate of an electrowetting microfluidic device. The embedded sensor had the highest reported figure-of-merit for any microresonator integrated with electrowetting microfluidics. The embedded microresonator sensor was also the first fully-embedded microresonator in an EWD system. Because the sensor was embedded in the top plate, full functionality of the EWD system was maintained, including the ability to move droplets onto and off of the sensor and to address the sensor with single droplets. Furthermore, the highest figure-of-merit for an SU-8 microresonator sensor yet reported at a probe wavelength of 1550 nm was measured on a test device fabricated with the embedded sensor structure described herein. Optimization of the sensor sensitivity utilized recently developed waveguide sensor design theory, which accurately predicted the measured sensitivity of the sensors. Altogether, the results show that embedding of a microresonator sensor in an EWD microfluidics system is a viable approach to develop a portable diagnostic system with the high efficiency sample preparation capability provided by EWD microfluidics and the versatile sensing capability of the microresonator sensor.
Dedication

To family and friends.
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1. Introduction

1.1 Motivation

1.1.1 Thesis Statement

Infectious diseases present a pernicious threat to the health of the world’s population, with more than 10.5 million deaths a year, over a million of which are caused by malaria [1, 2]. Multi-drug resistant tuberculosis and sleeping sickness (trypanosomiasis) are also important disease targets for new diagnostics, according to the Foundation for Innovative New Diagnostics (FIND) (http://www.finddiagnostics.org).

Portable diagnostic biosensors for resource poor environments have the potential to revolutionize medicine in the third world, however, the realization of these systems, especially for low cost systems, pose serious engineering challenges. These challenges include the need for low limit of detection, miniaturization, and system integration in a small, portable format, packaging the system for harsh environments, minimizing power consumption, using the lowest cost materials and manufacturing processes to realize a cost effective system, and achieving accurate and rapid results. The intersection of these eight engineering goals is being pursued by a number of researchers using a wide variety of sensing modalities. To date, no single modality has risen above the rest with clear advantage for field implementation. This thesis explores the intersection of these engineering system goals through the development of an optical
biosensing platform using optical microresonators integrated with a fluidic processing sample preparation system.

An important question to address for a portable diagnostic device is: What is the limit of detection that is sufficient for the application? In essence, to achieve low cost, low power, portable systems, significant engineering design tradeoffs must be made, and it is clear that the most sensitive biosensing lab techniques available today are large, power hungry, and difficult to manufacture and package. Microresonators have been used to detect DNA as well as other biomolecules. Therefore, the results presented in this thesis could be used to inform a portable DNA detection system. One way to determine how microresonator sensor technology applies to a portable diagnostic device is to explore a particular biosensing target, such as detection of malaria in blood by DNA markers.

1.1.2 Case Study: Malaria

An important global target for resource poor environments is malaria, which can be sensed using DNA in blood samples. Malaria, meaning ‘bad air,’ the name given for infections caused by the parasite family *Plasmodium* (*P*.), is one of the most widespread of infectious diseases and primarily affects only developing regions. Five distinct species of *Plasmodium* infectious to humans exist: *P. falciparum, vivax, ovale, malariae,* and *knowlesi.* Out of these five, *P. falciparum* is the most deadly. Although malaria has been eliminated in the developed regions of the world, it remains endemic to developing
regions in Africa, Central and South America, and Asia in strains resistant to commonly available drugs. Fifty percent of the world’s population is currently at risk for contracting malaria, and as climate change causes an increase in temperatures across the world, this risk is expected to increase [3, 4]. By 2020, the number of malaria cases is expected to at least double in the absence of effective interventions [5, 6]. Innovative disease diagnostics are an integral part of the World Health Organization’s (WHO) strategy to reduce and to eliminate the burden of malaria in the developing regions of the world [7], and is one of the motivating applications for the research proposed herein.

The 2008 Global Malaria Action Plan (GMAP) presents a detailed strategy for intervention, control, and elimination of malaria and specifically cites the need for “...tools that minimize the risk of emergence of drug-resistant malaria.” [7] In order to be useful for diagnosing diseases in the developing world, an innovative diagnostic device must be simple, requiring little to no training to use, portable, low-power, and inexpensive with a per-test cost of around US$1. Current inexpensive, rapid diagnostic tests (RDTs) for malaria consist of a dipstick type immunochromatographic assay that detects surface proteins called antigens that are present on the surface of *P. falciparum* and *P. vivax* parasites. They utilize antibodies to bind the cells in the sample, which is usually sputum or blood, to a substrate that transduces the molecular binding signal to a color coded result. The target antigens are primarily histidine-rich protein 2 (HRP-2) and *Plasmodium* lactate dehydrogenase (pLDH) [8]. However, although antigen tests
have been shown to be highly specific and sensitive, they cannot distinguish between
drug-resistant strains or between an active or inactive parasite infection. Furthermore,
RDTS based on antigen-antibody binding detection are only reliable if the blood content
of parasite, the parasitaemia, is greater 100 parasites/μL, or about 9 days after initial
infection in the case of *falciparum* malaria [8, 9].

Another way to detect the presence of infectious diseases in blood is by genetic
identification. Genetic diagnostics provide a means to assess not only the species and
subtype of an infectious disease, but the drug resistance as well. The ability to detect
drug resistance enables more judicious use of drugs to reduce the emergence of drug
resistance and also enables the detection of subpatent infections. Recent findings from
genetic microarray assays have shown that genetic markers can be used for diagnosing
drug-resistance. *P. falciparum* has developed widespread resistance to conventional
antimalarial drugs, such as chloroquine, Fansidar (sulfadoxine–pyrimethamine), and
amodiaquine. Chloroquine resistance and susceptibility to other heme-binding
antimalarials is conferred by mutations in the chloroquine resistance transporter (*pfcrt*)
gene, while resistance to sulfadoxine/pyrimethamine is conferred by mutations in
dihydropteroate synthetase (*pfldhps*) and *pfldhpr* genes [10, 11]. Mutations in the *pfmdr1*
and *pfldhps* were found to be reliable predictors of negative treatment outcomes with
amodiaquine+sulphadoxine-pyrimethamine combination therapy in Papua-New Guinea
[12]. Some resistance markers, such as those for chloroquine, work well across
populations, as was demonstrated in a rapid, real-time PCR assay developed for screening returning travelers for chloroquine resistant strains of *P. falciparum* [13]. In general, many factors besides genetic predisposition for drug resistance can affect clinical outcomes in treatment with a particular single or combination drug therapy, and resistance markers can vary between distinct host populations. Therefore, association studies between resistance markers and clinical treatment outcomes are necessary to validate resistance markers. Resistance markers also vary with time. Therefore, continuous monitoring of marker genes is needed to keep diagnostics up-to-date. Emerging, rapid, whole genome microarray assay technologies will enable tracking of changes in time and variations between populations of genetic markers [11]. Clinical diagnostic devices will be able to utilize this genetic information to accurately test for genetic markers of drug resistance by updating their DNA probes as the genetic markers vary. The ability to accurately test for drug resistance will help to prevent misapplication of treatments in individual patients and to monitor and prevent the development of drug resistance [14]. Thus, detection of drug resistance is a strong motivating factor for the development of DNA-based clinical diagnostics using DNA sensors.

### 1.2 Point-of-Care Device Requirements

Guided by the malaria example, some generalized requirements for a DNA-based biosensor can be established. The ideal biosensor would have a low enough LOD
to detect down to single molecule concentrations of DNA, proteins, enzymes, or cells with perfect specificity for the intended sensing target. A blood sample of 100 μL can contain 10 or less target molecules for a specific diagnostic, and sometimes none at all. As a demonstrative example, the parasite density, or parasitemia, on the sixth day of infection of *falciparum* malaria was measured to be approximately 0.012 parasites per microliter of blood [15]. Most biosensors reported to date cannot achieve single-molecule detection. For the few that can [16, 17], there remain significant problems for integration into a portable device.

To leverage low cost technologies for biosensing, considering the small quantities of target molecules expected in clinical samples, a means to amplify the sensor signal must be employed. For DNA, the polymerase chain reaction (PCR) is a robust process for increasing the quantity of DNA with a particular sequence [18]. PCR can achieve amplification of about $10^9$-fold in slightly more than an hour and has been demonstrated to enable detection of a single copy of DNA [19-21]. More recently, isothermal DNA amplification techniques have been developed [22]. Isothermal PCR is interesting for portable systems, because it eliminates the need for power hungry temperature cycling operations. One particularly well-studied example of isothermal PCR, called loop-mediated isothermal amplification (LAMP), achieved $10^9$-fold amplification in less than an hour [23]. Notably, a particular DNA test using this amplification technique was estimated to cost ~$0.50 per test [24].
Given that a wide variety of chemical amplification techniques are available for DNA and other proteins, an effective approach to developing a low-cost, portable biosensing platform would be to combine an inexpensive, lower LOD sensor technology with chemical amplification techniques. The feasibility of performing PCR in a microfluidic system has already been demonstrated. Both standard PCR and isothermal PCR processes for chemically amplifying the quantity of DNA have been implemented in microfluidic platforms [19-21, 25, 26]. The need for chemical amplification as well as sample preparation motivates the integration of this sensor technology with a microfluidic platform, which can perform these operations.

1.3 Biosensing Technologies

To determine what type of sensor technology would be best for a portable diagnostic system, it is necessary to compare different types of biosensing technologies. Biosensing technologies for the detection and identification of DNA have been demonstrated with a wide variety of different schemes. The field of DNA sensing is so vast and varied that a comprehensive review is beyond the scope of this thesis. Rather, a brief overview of the important points will be covered. All of the technologies can be roughly partitioned into three groups: electrical, mechanical, and optical. Many comprehensive reviews are available in the literature, including those for electrochemical sensors [27-29], mechanical sensors [28, 30-32] and optical sensors [33-35].
1.3.1 Electrochemical Sensors

A wide variety of techniques exist to detect DNA hybridization via an electrical signal [29]. Nearly all electrochemical sensors share two common features, (1) that DNA hybridization takes place on a conductive surface (except for the nanogap electrode system) and (2) that the sensor readout is obtained by either a current or a voltage measurement. Several sensors with LODs below 100 fM have been demonstrated [36-44]. Examples of electrochemical sensors include a redox polymer (polyacrylamide (PAA)-poly(4-vinylpyridine) (PVP)-[Os(bipyridine)2Cl]+/2+) and DNA probe-modified glassy carbon electrode with enzyme amplification by bilirubin oxidase [36], an enzyme amplified probe-modified carbon nanotube sensor [42], a field effect transistor-based (FET) sensor [45], a probe-modified gold electrode with amplification by ‘biometallization’ [39], and a nanogap electrode with amplification by ‘bio-bar code’ and metallization of bound target DNA in the electrode gap [38]. Several reports indicate detection limits in the thousands of molecules or less. Several electrochemical sensor technologies are commercially available, including the CombiMatrix™ ElectraSense®, the GenMark Dx™ eSensor®, and the eBiochip GmbH eMicroLISA®. Notably, the ElectraSense® system supports arrays of up to 180,000 sensor elements. The main drawback of electrochemical sensors is that they require many chemical amplification steps to achieve LODs competitive with the other two sensing technologies.
1.3.2 Mechanical Sensors

Examples of mechanical sensors include quartz crystal modulators (QCM) [46], microcattilevers [47], surface-acoustic wave (SAW) devices [48], and thin-film bulk acoustic resonators (FBAR) [49]. DNA at a concentration below 100 fM has been detected with QCMs and microcantilevers [46, 47, 50]. Commercial sensors include the Initium Affinix® (a QCM sensor) and the Attana Attana™ (a QCM sensor). The main drawbacks of QCMs are relatively high fabrication costs and limited sensor arraying capability. For mechanical sensors in general, mechanical stiction that occurs in liquid media poses a significant challenge to obtaining high performance biosensing [51].

1.3.3 Optical Sensors

Sensors with LODs below 100 fM include [52-62]. Examples of optical sensor technologies include fiber optic array fluorescence sensors [61], direct fluorescence measurement systems [56], surface plasmon resonance (SPR) sensors [52], and microresonators [63]. There are several commercialized technologies for optical sensors. These include SPR from GE Healthcare (Biacore) and Biosensing Instruments (BI), SPR imaging from GE Healthcare (Flexchip), optical gratings from Corning (Epic), far-field interferometry from Farfield (Analight), bio-layer interferometry from ForteBio (Octet), ellipsometry from Maven Biotechnologies (LFIRE), nanopore optical interferometry from Silicon Kinetics (SkiPro), and direct fluorescence from Affymetrix (GeneChip® Scanner). The drawbacks of optical sensing technologies are the relatively high cost of
optical sources and detectors as compared to comparable electronic sources and detectors. Additionally, fabrication of non-planar optical structures is costly and integration with sources and detectors via external coupling is not power efficient.

1.3.4 Comparison of the Sensing Technologies

From the foregoing brief survey, it is clear that technologies from all three primary classes of DNA sensors have seen some commercial success. Table 1 compares the advantages and disadvantages of the three primary sensor classes.
Table 1. Advantages and disadvantages of the main types of DNA sensors.

<table>
<thead>
<tr>
<th>Type</th>
<th>Advantages</th>
<th>Disadvantages</th>
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| Electrochemical | • Low-cost fabrication  
• Low-cost readout  
• Sensitive to electrical signals  
• Commercially available  
• Supports very large arrays  
• Integration of sources and detectors is possible | • Chemical amplification steps are required to achieve the lowest detection limits |
| Mechanical    | • Low-cost readout  
• Sensitive to mass  
• Insensitive to electrical signals  
• Integration of sources and detectors is possible  
• Commercially available | • Stiction reduces sensitivity in liquid media |
| Optical       | • Detection limits down to single-molecules without labeling  
• Low-cost fabrication for planar structures  
• Sensitive to mass  
• Insensitive to electrical signals  
• Supports very large arrays  
• Commercially available | • High-cost and difficult readout  
• High-cost fabrication for non-planar structures |

On the basis of this comparison, electrochemical sensors are strong candidates for low-cost, portable lab-on-chip clinical diagnostic systems in the near term, but suffer from LOD limitations; Although challenges remain for low cost and power efficient readout.
of the optical signals, optical sensors have the greatest potential for low limit of detection and miniaturized, integrated arrays of sensors in a low power format.

### 1.3.5 Advantages of Optical Sensors

There are several advantages of optical sensors compared to the other technologies. Of the three primary sensing technologies, optical systems provide the lowest limits of detection without target labeling, down to single-molecules [16]. With appropriate implementation of design and fabrication techniques, such a low limit of detection may also be achievable with planar device structures when combined with chemical amplification. Optical systems measure changes in the polarizability density (mainly the density of electric dipoles) of the materials with which a probe light signal interacts, a property that is inherent to any material made up of atoms, charged or uncharged, conductive or non-conductive. The refractive index is an effective medium property that is related to the polarizability density of the material and is the optical property typically specified for a material that defines its interaction with light waves. Thus, optical sensors provide complementary measurement information to that of electrochemical sensors. Furthermore, because optical sensors can detect materials independent of conductivity and charge properties, it is relatively straightforward to apply them to other target biomolecules of interest for clinical diagnostics, such as proteins and antibodies.
POC diagnostic systems are typically designed to have a very low cost disposable component, which handles the human samples. The component that contacts the samples is disposable to address contamination and biofouling. Except for photodetectors, these sensors must be located within the microfluidic channels of the disposable component and, therefore, must themselves be disposable. The disposability of the sensor is, therefore, a key consideration in the selection of a sensor technology. Most sensors contain inorganic materials, which are costly and pose a toxicity risk when disposed [64]. Organic materials do not offer the refractive index contrast available in inorganic materials (which limits the sensor performance). However, organic materials are lower cost and simpler to process than inorganic materials, and many are biocompatible [64].

The main challenge to overcome for optical sensors, besides optimization of the sensor performance, is the measurement of an optical signal with a low cost, small sized, and power efficient readout system. For systems in which a signal is measured as a change in power in a narrow band of wavelengths, the readout can be done with well established technology, such as with a light-emitting diode or a laser diode as an optical source and a photodiode as a detector. However, power measurements are highly susceptible to noise [65], so noise rejection schemes, such balanced detection with lock-in amplifier, must be employed [66]. Also, when using power measurement as a readout for measuring shifts of resonances in the spectrum of a resonant optical sensor, a type of
sensor that is typically used to detect refractive index changes, the response is generally nonlinear. Because of these problems with narrow band or single wavelength power measurements, resonant optical sensors are most often measured by making power measurements at a series of different wavelengths, which, as a group, comprise a spectrum (power versus wavelength) measurement. For the current gold standard of refractive index optical sensors, surface plasmon resonance (SPR) sensors, it is feasible to utilize a dispersive optical element combined with an array of photodetectors to measure the spectrum and determine the wavelength (or angle) corresponding to the SPR resonance minimum [67]. For resonances produced by high quality (Q) factor (Q>10^4) cavities (i.e. those that have very sharp resonances), such as a microresonator, the spectral resolution of dispersive elements is typically inadequate to resolve the resonances, or to obtain the maximum resolution in the estimate of the resonance peak or null wavelength [65]. Also, for high-Q factor resonant cavities, if the power measurement approach is to be utilized, the probe light must have a very narrow bandwidth to be effective, because the resonances of high Q resonant cavities are less than about 150 pm for Q factors in excess of 10^4. For these reasons, spectral measurements of high Q resonators are typically performed with a tunable laser with a linewidth significantly smaller than the sensor’s resonance linewidth.

Recent developments in laser technology may enable integration of a high performance tunable laser system in a small, portable package. Particularly, the discrete
mode (DM) laser is a very promising laser technology for portable tunable laser applications as well as single-wavelength power measurements for measuring resonant shifts in high Q resonators. DM lasers are fabricated as low cost, Fabry-Perot laser diode chips, achieve very narrow linewidths (on the order of 1 fm at 1546 nm), and can be temperature tuned over a range of about 1-2 nm [68]. DM lasers are currently commercially available from Eblana Photonics in a wide range of wavelengths as well as in custom wavelengths. Such narrow linewidth lasers are key to the practical implementation of optical resonant sensor technologies as components in portable sensing platforms. Thus, it is possible to envision a compact, portable optical readout system with a DM laser externally coupled into the disposable optical sensor system and with the output optical power measured by a photodetector that is either internally coupled to the optical sensor system for improved coupling efficiency or externally coupled to the optical sensor system for reduced cost of the optical sensor system.

1.4 Proposed Diagnostic System

Microresonators have been identified as strong candidate optical sensors for POC diagnostic sensing applications and have been extensively investigated. Of particular interest is polymer microresonators, which can be fabricated at low cost, but with a performance penalty relative to inorganic material based microresonators. Thus, design of the microresonator is critical to minimize the performance gap. Additionally, for this sensor to be useful in POC applications, it must be integrated with a microfluidic sample
preparation system. This thesis addresses these issues by focusing on the design and optimization of a planar microring resonator and its integration with a digital microfluidic system. This combination of technologies has the potential to produce a small-form factor, low-power, portable system with integrated sample preparation and optical sensing.

The diagnostic device envisioned will consist of a durable and reusable component housing a disposable sample handling component. The reusable component will contain the display, signal processing, control, and power electronics as well as the optical readout to limit the cost of the disposable device as much as possible. The disposable component, in which the samples are handled and sensing occurs, will be changed for each test. The disposable component will contain a ‘digital’ (droplet-based) electrowetting-on-dielectric (EWD) microfluidics system [69] to perform sample preparation, including filtering and PCR amplification, and sensing. The sensing element to be integrated with the EWD system will consist of a low-cost, polymer-based, planar, vertically-coupled microring resonator [70].

1.5 Research Objectives

The objective of the work described herein was to develop a biosensing platform with an optical microresonator biosensor integrated with an EWD sample preparation system. The objectives given below summarize the specific goals of the work:
1. Design and optimize a polymer microresonator sensor with buried bus waveguides designed for integration with an EWD system.

2. Design and demonstrate sensing with an optical microresonator sensor integrated into the top plate of a droplet-based ‘digital’ EWD microfluidic system, such that the operation of the EWD system is unaffected by the sensor.

3. Compare theoretical performance parameters to the experimentally measured performance parameters.

1.6 Background and Justification

There is a need for portable, point-of-care (POC) clinical diagnostics to diagnose diseases using complex assays, such as those utilizing DNA identification, both for treating diseases in the developing world as well as for personalized healthcare [71]. POC diagnostic systems must have several attributes, including low cost, low power consumption, small size, low reagent consumption, high accuracy, rapid time-to-result, and ruggedness [71]. Several components are necessary, including a sensor to detect biomolecules, a microfluidic system to perform sample preparation, a ‘world-to-chip’ interface, control and interface electronics, power electronics, and packaging [71]. In terms of sample preparation, POC systems specifically require the ability to extract target molecules, such as DNA or proteins, from complex samples, such as blood. A wide variety of microfluidics platforms [71-73] as well as sensor technologies [27-35] have been developed to address the needs of sample preparation and of analyte
detection, respectively. All of the POC device components would ideally be packaged into a device about the size of a cellular telephone and would be powered by watch batteries. A very low cost disposable component would handle the human samples and perform the sensing. One of the major challenges to the realization of a POC device capable of handling complex immunoassays, such as identification of pathogens and their drug response by DNA identification, is the integration of the sensor into a microfluidic device that would maintain all of the desired attributes for a POC device.

Microfluidic platforms can be categorized as either continuous flow or droplet-based flow systems [72, 74]. Pressure driven continuous flow systems are appealing and are used extensively in laboratories and in some commercial devices, because they are both inexpensive and easy to fabricate. Sample preparation from complex human samples, such as blood, has been demonstrated with these systems [75]. However, pressure driven systems require bulky external pumps that consume a large amount of power and contain a relatively large amount of so-called ‘dead volume’, which is fluid that flows through the channels in the system that performs no other useful function besides filling space in the flow channels [72, 73]. Droplet-based flow systems, in which fluids are driven in the form of separate plugs or droplets, naturally have much less dead volume than continuous flow systems. The reduced dead volume results in reduced reagent usage and more efficient use of the limited clinical sample material. Droplet-based flow systems can perform complex assays involving sample preparation
from complex samples, like continuous flow systems with very low power and without the need for bulky external pumps [73, 76]. Therefore, droplet-based flow systems satisfy the criteria of low power, low reagent consumption, and small size. These attributes motivate research on the integration of sensors with droplet-based flow systems.

There are three major types of droplet-based flow systems, including segmented flow, a type of pressure driven technology, electrowetting-on-dielectric (EWD), a type of electrokinetic technology, and surface acoustic wave, a type of acoustic technology [72, 73]. Segmented flow devices, while well established, are pressure driven and so do not bring all of the advantages of the other types of droplet-based flow. Surface acoustic wave devices are distinguished by their flexibility in that droplets of fluid can be actuated regardless of their polarizability or ion content. However, the power required to transport droplets is relatively high (watts to milliamps of power) [77-79]. EWD, while affected by the polarizability and ion content of liquids, has been shown to be capable of moving a variety of different types of fluids down to tens of picoliters in volume [80]. EWD also has the additional advantage of reconfigurability. Furthermore, EWD has been demonstrated for sample preparation, including extraction of DNA from a blood sample and amplification of the DNA with PCR [25]. Two other notable droplet-based flow technologies, optoelectrowetting and magnetic droplet actuation, may potentially prove useful in the future, but are still in early stages of development.
Among droplet-based flow technologies not requiring external pumps, EWD is arguably the most well developed and is a very promising microfluidic platform for a POC diagnostic device.

An important aspect of droplet-based flow systems is the need for an immiscible gas or liquid medium to surround the droplets of fluid. For systems with enclosed flow channels, liquid media are most commonly used and include silicone oil, dodecane, and hexadecane [81]. The medium performs several functions, including enabling the formation and movement of individual plugs of fluid in segmented flow systems, preventing evaporation, and preventing surface fouling [72, 76]. In the particular case of EWD, the immiscible medium also serves the important function of reducing the threshold voltage for droplet actuation, which increases the device reliability [76]. While it is possible to fabricate EWD devices that can actuate droplets in an air medium, devices which are somewhat more demanding in design and fabrication [82], the use of a liquid medium is preferred, due to the previous reasons mentioned as well as to the reduced fabrication complexity/cost. Two potential problems are readily apparent for the integration of surface sensors with a droplet-based flow system utilizing a liquid medium. The first is the effect of the immiscible liquid medium on the ability of biorecognition chemistry to occur on or near the sensor surface. The second is the effect of the liquid medium on the sensor’s limit-of-detection (LOD). The first problem has received little, if any, attention, and the latter problem has only recently begun to be
addressed [83, 84]. Experiments involving glucose sensing with a microresonator
integrated with an EWD system by Luan, et al. showed that a microresonator sensor
remained functional in the silicone oil medium [83, 84]. However, the effect of the
silicone oil on sensor performance was not quantified. Therefore, more research is
necessary to characterize the effect of the immiscible medium on surface sensor
performance and on what steps can be taken to mitigate the any deleterious effects of
this medium. The results of this research have implications not only for surface sensor
integration in EWD microfluidics, but more generally for surface sensor integration in
microfluidic platforms that utilize a liquid immiscible phase as well.

Because the advantages conferred by a liquid immiscible medium cannot be
ignored, the performance of any sensor integrated with a flow system having a liquid
immiscible medium must be evaluated in the presence of this medium. Many highly
sensitive biosensors reported to date transduce a signal produced by a change in a
material property on or near the surface of the sensor (surface sensing). These sensors
can be divided roughly into three categories: electrical [27-29], mechanical [28, 30-32],
and optical [33-35]. Salient examples of these types of sensors include the
microcantilever [85], a type of mechanical sensor, surface plasmon resonance (SPR) [86]
and the microresonator [87, 88], both types of optical sensors, and electrochemical
impedance spectroscopy [89] and the ion sensitive field effect transistor (FET) [90], both
types of electrical sensors. Surface sensing sensors are important not only because they
are highly sensitive in the low limit of detection (LOD) sense, with some able to detect single molecules [16, 91], but because many of these sensors are planar devices, which can be fabricated with low cost, well-established planar fabrication technology [92]. Furthermore, surface sensors are capable of unlabeled detection, which is the detection of biomolecules without the need for additional molecular labels, such as fluorophores for optical detection or redox tags for enhanced electrochemical detection.

Optical sensors are particularly attractive for integration with EWD systems, because they can sense materials in a droplet surrounded by a liquid immiscible medium and because they are insensitive to electrical phenomena. Fluorescence, chemiluminescence, and absorption sensing are some of the most common and straightforward ways to perform immunoassays [64, 71]. These methods of sensing are particularly appealing, because the sensing element, the photodetector, does not need to be inside the fluid channel, avoiding the problems of surface fouling, non-specific binding, and any performance degradation due to immersion in the immiscible medium altogether. In fact, a detection limit as low as a single molecule using fluorescence with integrated microfluidics has been demonstrated in a relatively small, packaged system [93]. However, integrated free space optical measurement at present requires bulky filter sets and high voltage avalanche photodetectors (APDs), which limit the minimum device size. Optical surface sensors have advantages over fluorescence detection in terms of device size (bulky external elements are not necessary) and design complexity,
as well as the capability to be fabricated in dense arrays for the detection of multiple targets in a small package [94]. Some notable examples of optical surface sensors integrated with microfluidic systems are the liquid core microresonator [95] and SPR [96, 97] and microresonator sensors [94] integrated with pressure driven flow microfluidic systems. A thorough review of opto-fluidic technologies by Brennan, et al., provides an insightful comparison of the trade-offs between these and other types of integrated optical sensing technologies [64].

There are two particular advantages of optical surface sensors in the EWD system over other types of surface sensors. First, because optical sensors detect changes in the refractive index of media for a significant distance above the sensor surface, due to the extension of the guided optical wave’s evanescent tail outside of the waveguide core, which is typically in the range of 100 nm to 1 μm for probe wavelengths near 1550 nm, a thin film of immiscible liquid medium coating the surface of the sensor would not be expected to completely eliminate sensor functionality if its thickness is smaller than the extent of the evanescent tail. Second, because optical sensors are insensitive to electrical charge, they are not susceptible to the electrical charge-based phenomena encountered in the EWD system [98]. Due to this second factor, optical sensors may be more suitable for integration with EWD microfluidics than electrical sensors. On this basis, mechanical sensors, such as the microcantilever sensor and the surface acoustic wave
sensor, could also be equally suitable for integration with EWD microfluidics as they are also insensitive to electrical charges.

Optical surface sensors are considered to be one of the most straightforward devices to implement for performing unlabeled detection by detecting changes in refractive index [64, 71]. Probably the reason that this is so is because of the success of SPR as a refractometric sensor for characterization of surface binding in laboratory settings. SPR has been commercialized in several products, including GE’s Biacore and Biosensing Instrument’s BI series of SPR sensors. SPR is a particular type of sensor that belongs to a larger class of waveguide refractive index sensors, which also includes the Mach-Zehnder interferometer (MZI) [99], the grating-coupled waveguide [100], the fiber optic sensor [101], the Fabry-Perot resonator [102], and the microresonator [94]. In waveguide refractive index sensors, refractive index is typically measured by using a waveguide combined with a second optical component to transduce the change in the phase constant of the guided mode of the waveguide to a measureable signal proportional to the change in refractive index near the surface of the waveguide [34]. Given the success and proven capability of SPR sensors, they would seem to be the optical sensor technology of choice for integration. However, waveguide SPR demonstrations are only beginning to emerge and do not yet perform as well as commercial SPR devices [34, 103, 104]. Additionally, precious metal coatings on waveguides are highly lossy and are not a low cost materials suitable for a disposable
part. Commercial SPR sensors are generally characterized by very high sensitivity, but large resonance linewidth (low Q factor). The combination of high sensitivity and low Q in commercial SPR systems typically yields a refractive index limit of detection of $10^{-5} – 10^{-7}$ RIU [64]. Microresonator sensors have been shown to achieve detection limits comparable to commercial SPR sensors [65], very low optical attenuation, and a high Q factor and have been demonstrated in a waveguide format. Furthermore, planar microresonator sensors can be fabricated using low cost polymer materials, whereas SPR sensors typically utilize gold or silver, and in a planar geometry, enabling use of low cost planar fabrication techniques [92].

The work presented herein advances the state of the art for planar optical microresonator sensor integration with a microfluidic platform capable of performing sample preparation with efficient use of reagents and with low power, electrowetting-on-dielectric (EWD). Specifically, advancements were made both with the microresonator sensor design and with the performance of the integrated microresonator sensor – EWD system. An accurate theory of design was developed and implemented to improve the sensor sensitivity. Using this theory, a vertically-coupled polymer microresonator sensor was developed that achieved a sensitivity (S) of 82 nm/RIU and a quality (Q) factor of 15,000, yielding a figure of merit (FOM), the product of Q and S, of $1.2 \times 10^6$ nm/RIU. The FOM as defined by the product of Q and S is a useful figure for comparing the performance of microresonator sensors, because the intrinsic
limit-of-detection (LOD) of a microresonator sensor is inversely proportional to this FOM [65]. This measured FOM is comparable to that of other polymer microresonators, including those made with polystyrene (1*10^6 nm/RIU at 1550 nm) [105], SU-8 (2.3*10^6 nm/RIU at 1310 nm) [106], and ZPU13-430/LFR-S708U (2*10^6 nm/RIU at 1550 nm) [87]. Furthermore, this FOM is the highest reported for an SU-8 microresonator probed around 1550 nm.

In addition to the development of a well-performing polymer microresonator using design methodology, the EWD-microresonator sensor integrated system performance was improved in several ways compared to previously reported work. The EWD system integrated microresonator sensor had a nominal sensitivity of 89 nm/RIU and a sensitivity of 72 nm/RIU in silicone oil, a Q factor of 8,400 in water, and the same Q factor in the EWD system. These parameters translate to a nominal FOM of 0.75*10^6 and an EWD integrated FOM of 0.60*10^6. The EWD integrated FOM of this device was nearly double that of the previously reported SU-8 microresonator sensor integrated with an EWD system [84]. As for the performance of the integrated system, several improvements were made. In the previously demonstrated system, a large quantity of liquid had to be dispensed onto the sensor in order to make contact with the sensor through a via hole in the top plate. Additionally, water could not be removed from the sensor once applied, because it was wetted to the walls of the via. In this work, the microresonator sensor was embedded in the top plate, such that the effect of the sensor
integration on the microfluidic actuation was greatly reduced. The sensor was addressable with a single droplet and the ability to move droplets onto and off of the sensor was demonstrated. Finally, in addition to the performance improvements, the effect of silicone oil on the sensitivity of a microresonator sensor was quantitatively evaluated for the first time to this author’s knowledge.
2. Theory

2.1 Optical Waveguide Circuit Layout

The optical system utilized in this work consisted of several components, including a circular microresonator, two waveguides to which it is coupled, and three fiber-waveguide coupling regions. The diagram in Figure 1 shows the most recent iteration of the design of the optical system with all of the important components labeled.
Figure 1. Optical system diagram and optical spectra as measured at each port.

To provide an understanding of how this optical device functions, it is important to understand the path of a light wave as it travels through the system. Light generated from an optical source, such as a laser, is coupled into one end of an optical fiber. The cleave at the opposite end of the fiber is aligned to the facet of the input waveguide. After the light exits the fiber, it couples into the input waveguide, which is intentionally
wide to more efficiently collect the input light. The light travels through the input waveguide until it reaches the input taper. As the light passes through the input taper, it is squeezed into the smaller throughput waveguide. After the input taper, the light travels to the throughput coupler. At the throughput coupler, a fraction of the light couples into the microresonator waveguide, or microresonator, and the rest continues on to the throughput port. The light that was coupled into the microresonator will, on average, circulate in the microresonator for a time \( \tau \), the cavity lifetime, before exiting at either the throughput coupler or the drop coupler. The light coupled out of the microresonator at the drop coupler travels through the drop waveguide until it reaches the drop port. At both the throughput and the drop ports, the light is collected by an optical fiber, which is aligned to either the throughput port or the drop port facet. The output light travels through the fiber into a photodetector and its power is measured.

The recirculation of light in the microresonator produces spectral features that can be observed if the laser wavelength is tuned across a range of wavelengths and power is measured at many points throughout the scan as shown in Figure 1. One of these features is depicted in Figure 2. These features are the result of light resonating in the microresonator. The wavelengths of these resonances are very sensitive to the environment near the surface of the microresonator. By tracking changes in the resonant wavelengths, one can measure changes in the environment near the microresonator surface.
2.2 System Performance Parameters

Fundamentally, a sensor’s performance is defined by the smallest quantity of something that it measures and is known is the limit of detection (LOD). For a microresonator sensor, the LOD is related to three performance parameters intrinsic to the optical system, including the microresonator, the bus waveguides, and the input/output coupling ports: the quality factor (Q), the sensitivity (S), the insertion loss (1-\(T_{in}\)), where \(T_{in}\) is the insertion transmission.

The quality factor (Q) is equal to the ratio of the energy stored to the energy lost per round trip in the microresonator and is a measure of the sharpness of the resonances. The sharpness of the resonances affects the estimation of the resonant wavelength in measured spectra by effectively filtering intensity noise [65]. A sharper resonance leads to a higher wavelength resolution by providing better filtering of the intensity noise. The Q is determined by the optical losses in the microresonator as well as the coupling loss from the microresonator to the bus waveguides. The sensitivity (S), usually given in nm/RIU (RIU = refractive index unit), quantifies the microresonator spectral shift due to changes in the refractive index near its surface. S is an intrinsic property of the microresonator and is affected by the cross-sectional dimensions and the refractive index profile of the microresonator waveguide and cladding materials. The insertion loss is defined as the total loss of optical power from input to output in the optical system.
Figure 2. A hypothetical resonance spectral feature as it would be observed from the
from the throughput port spectrum and from the drop port spectrum. Important
parameters are labeled. (Left) Throughput port (see equation (20) for the throughput
FWHM definition). (Right) Drop port.

Insertion loss is an important parameter for system design, because the signal-to-
oise (SNR) ratio of the individual power measurements made in a spectral
measurement of the microresonator is affected by the insertion loss. Two problems can
occur with high insertion loss: either the measurement precision will decrease, due to
the photodetector noise being large relative to the signal, or no signal will be
measureable (signal less than the photodetector noise floor).

Q and S are important to the system design, because the ultimate refractive index
limit of detection of the optical system is inversely proportional to a figure of merit
(FOM), which is equal to the inverse of the product of Q and S [65, 107]. Insertion loss is
not included in this FOM, but the effect of insertion loss on the LOD is significant and
can limit the maximum Q factor of the device. Because both the Q factor and the
insertion loss are dependent on the degree of coupling between the resonator and the
bus waveguides, there is a trade-off between Q factor and insertion loss for each measurement port. A larger coupling gap decreases the bus waveguide-microresonator coupling, which increases the Q factor, reduces the insertion loss for the throughput port, and increases the insertion loss for the drop port.

On this basis, the throughput port would generally provide the best performance for sensing by measuring the shift in resonant wavelengths in the case of weak coupling, having both high Q and low insertion loss. However, there is a maximum limit to the decoupling of the microresonator from its bus waveguides to be able to detect a resonance at the throughput port spectrum. This limit is set by the detector noise and the extinction ratio (ER), the ratio between the on-resonance and off-resonance transmission. As the microresonator becomes more decoupled from its bus waveguides, the extinction ratio at the drop port increases and that at the throughput port decreases. In order to track a resonant spectral feature, the difference between the power on-resonance and the power off-resonance should be at least three times larger than the detector noise level. Thus, the decision to use the drop or the throughput port depends on how Q, S, insertion loss, extinction ratio, and detector noise optimally balance for a particular measurement system. In lieu of such an analysis, it is common practice in the field to measure spectra from the most convenient port, or from whatever port provides qualitatively the best looking spectrum.
There are some specific cases in which the choice of measurement port is clear. In the case that the insertion loss at the drop port is high, such that the detector SNR affects the LOD more than other factors, spectra can be measured from the throughput port. In the case that the insertion loss is comparable at the throughput port and at the drop ports, then the drop port may be a better choice, because the resonant wavelengths manifest as maxima rather than minima, resulting in a better SNR in the drop port for the points around the peak, which are used to estimate the resonant wavelength. In the case that the extinction ratio is too small, it may be possible to obtain a better measurement of the spectrum at the drop port, which would have a much greater extinction ratio. To measure the Q factor, the drop port spectrum is usually the best measurement port, because the Q factor is defined more clearly for a peak than it is for a null corresponding to a wavelength of resonance. Finally, to determine the coupling parameters and internal loss of the microresonator for a microresonator that is asymmetrically coupled to two or more bus waveguides, it is necessary to measure spectra at both the drop port and the throughput ports. Whatever port is chosen for measurement, the insertion loss should be kept low enough that the SNR at the detector is not the limiting factor.

The following discussion will elucidate the connections between the three system performance parameters and the design parameters that were used to optimize the sensors reported in this work.
2.3 Waveguides

2.3.1 Waveguide Structures

Analysis of the guided modes of the waveguide modes of the system is necessary to assess the input/output coupling efficiency and to understand the trade-offs between the Q factor, the sensitivity, and the microresonator-bus waveguide coupling. The waveguide structures for the three types of waveguide in this system are shown in Figure 3.
Figure 3. Refractive index profiles and geometry of the waveguides in the optical system. (Top) Input waveguide. (Bottom) Throughput/Drop coupler. In the coupler diagram, the top waveguide is the microresonator waveguide, the bottom waveguide is the bus waveguide, and the material in between is the interlayer dielectric.

Accurate values for both the real and imaginary part of the material refractive indices are necessary for accurate calculations of the guided mode properties. For all
calculations and simulations, the parameters in Table 2 were used, unless specified otherwise.

Table 2. Material properties.

<table>
<thead>
<tr>
<th>Material</th>
<th>Waveguide Part</th>
<th>Symbol</th>
<th>n</th>
<th>n Ref.</th>
<th>k</th>
<th>k Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>SU-8</td>
<td>Core</td>
<td>(n_{\text{co}})</td>
<td>1.569</td>
<td>[108]</td>
<td>4.686*10^{-6}</td>
<td>[109]</td>
</tr>
<tr>
<td>H\textsubscript{2}O</td>
<td>Cladding</td>
<td>(n_{\text{cl}})</td>
<td>1.318</td>
<td>[110]</td>
<td>9.8625*10^{-5}</td>
<td>[110]</td>
</tr>
<tr>
<td>SiO\textsubscript{2}</td>
<td>Substrate</td>
<td>(n_{\text{sub}})</td>
<td>1.444</td>
<td>[111]</td>
<td>-0</td>
<td>[111]</td>
</tr>
</tbody>
</table>

\(^{1}\text{Varies depending on what region the sensed change in refractive index occurs. The region is always defined such that its refractive index is homogenous.}\)

The refractive index for SU-8 was determined using the Cauchy equation with parameters for SU-8 given the vendor datasheet [108]. The imaginary part of the refractive index for SU-8 was estimated from the propagation loss value of 1.65 dB/cm at 1550 nm measured for an SU-8 waveguide with a slightly lower refractive index SU-8 cladding with unpolarized light [109]. This value was selected, because it was measured with unpolarized light and the low refractive index contrast of the measured waveguide would have minimized the scattering loss component of the measured loss due to surface roughness on that waveguide. The complex refractive index of H\textsubscript{2}O has long been accurately characterized and was obtained from [110]. The same is true of amorphous SiO\textsubscript{2}, for which the refractive index of fused quartz was used [111]. However, the refractive index of SiO\textsubscript{2} films deposited by low temperature plasma-enhanced chemical vapor deposition (PECVD) is expected to deviate from this value.
2.3.2 Waveguide Modes

The concept of waveguide modes is important to understanding waveguides. A waveguide generally supports multiple guided modes in one of two different polarization states, either transverse electric (TE) or transverse magnetic (TM). As an example, Figure 4 shows the simulated mode profiles for TE modes for a 4 μm wide by 1.6 μm thick SU-8 waveguide embedded in SiO₂.
Figure 4. Normalized x-component (E_x) of the electrical field for TE modes of a 4 \textmu m wide embedded SU-8 bus waveguide overlaid with the waveguide cross section (black lines). The substrate, first top cladding, and second top cladding are SiO_2, SiO_2, and air, respectively. (a) TE00 (fundamental mode). (b) TE01. (c) TE02. (d) TE10.

From the mode profiles, one can see different numbers of nodes (nulls in the field profile) for different modes, and that in a waveguide with a 2-D profile, such as the one
simulated for Figure 4, these nulls appear in increasing number along both the lateral (x) direction and the vertical (y) direction. The guided modes of a waveguide are identified by the number of nodes in the lateral and vertical direction with the indices \( m \) and \( n \), respectively. For the fundamental mode, \( m \) and \( n \) both equal 0. To completely identify a mode, the polarization state and both the \( m \) and \( n \) indices must be given. TE modes are identified by \( \text{TE}_{mn} \) and TM modes are identified by \( \text{TM}_{mn} \). For example, the fundamental TE mode would be identified by \( \text{TE}_{00} \).

An important concept in understanding guided mode devices is polarization. In the context of a slab waveguide, which is defined to be infinite in extent in the lateral direction, the polarization of the guided modes is determined by which component of the electromagnetic field is perpendicular (transverse) to the direction of propagation. Solving Maxwell’s equations for the slab waveguide structure results in two orthogonal sets of solutions, each of which define the guided electromagnetic waves of the structure.
Figure 5. Field components and spatial orientation of the TE and TM polarized modes in a slab waveguide and a channel waveguide structure. Components in parenthesis are the minor field components.

In the coordinate system defined in Figure 5, one of these sets of solutions has only the field components $E_x$, $H_y$, and $H_z$, where $E$ denotes the electric field, $H$ denotes the magnetic field, and the subscript denotes the direction of the field component. These solutions are referred to as the transverse electric (TE) modes. Modes described by the other set of solutions, all of which only have the field components $H_x$, $E_y$, and $E_z$, are referred to as the transverse magnetic (TM) modes. The properties of the TE or TM fields can be completely described with knowledge of the $E_x$ or $H_z$ component, respectively.

For this and most other practical optical systems, slab waveguides are impractical. Usually, channel waveguides are used to route light to the desired destinations. In channel waveguides, pure TE and TM modes do not exist. Rather, the modes that closely resemble the TE and TM modes in channel waveguides are referred to as quasi-TE(TM) or TE(TM)-like. The TE(TM)-like modes in the channel waveguide
have a major component, which is made up of the field components corresponding to the slab waveguide TE(TM) mode, and a minor component, which is made up of the components corresponding to the slab waveguide TM(TE) modes. Figure 5 indicates the minor components for the TE(TM)-like mode in parenthesis next to the major components. Generally, a full-vectorial numerical mode solver must be employed to calculate the mode profiles and propagation constants in an arbitrary channel waveguide structure to account for all of the field components for each major polarization. For the particular case of the rectangular channel waveguide, the minor components are much smaller than the major components, which enables the use of the more efficient semi-vectorial numerical algorithm that ignores the contribution of these components [112, 113].

Polarization is important to the waveguide-coupled microresonator system, because all three performance parameters, insertion loss, quality factor, and sensitivity, are generally polarization dependent and often significantly so. Thus, both polarizations must be considered in the system design and polarization should be controlled during measurements.

**2.4 Insertion Loss**

Insertion loss is a term used to describe total power optical loss for an optical component. In this analysis, it is more straightforward to represent the insertion loss as a transmission, $T_{\text{ins}}$. The term insertion loss will be used from here forward to mean
either the transmission or loss. The insertion loss relates the input power to the output power for the optical system:

\[ P_{\text{out}} = T_{\text{ins}} P_{\text{in}} , \]  
(1)

where \( P_{\text{in}} \) is the input power and \( P_{\text{out}} \) is the output power. Insertion loss for the waveguide-coupled microresonator optical system is defined by the following equation:

\[ T_{\text{ins}} = T_{\text{in}} T_{\text{prop}} T_{\text{res}} T_{\text{out}} , \]  
(2)

where \( T_{\text{in}} \) is the input coupling efficiency, \( T_{\text{prop}} \) is the waveguide propagation transmission, \( T_{\text{res}} \) is the microresonator transmission, and \( T_{\text{out}} \) is the output coupling efficiency. This equation can be used for both the throughput and the drop ports. For the throughput port, \( T_{\text{prop}} \) is defined as \( T_{\text{prop,thru}} \), the propagation transmission in the throughput waveguide, \( T_{\text{res}} \) is defined as \( T_{\text{thru}} \), the microresonator throughput transmission, which will be defined in the following section, and \( T_{\text{out}} \) is defined as \( T_{\text{out,thru}} \), the throughput port output coupling efficiency. For the drop port, \( T_{\text{prop}} \) is defined as \( T_{\text{prop,drop}} \), the propagation transmission of the optical path from the input port to the drop port, \( T_{\text{res}} \) is defined as \( T_{\text{drop}} \), the microresonator drop port transmission, which will be defined in the following section, and \( T_{\text{out}} \) is defined as \( T_{\text{out,drop}} \), the drop port output coupling efficiency. Additionally, \( T_{\text{thru}} \) and \( T_{\text{drop}} \) are defined as the insertion loss for the throughput optical path and the insertion loss for the drop optical path, respectively. In order to characterize the optical system, in addition to measuring insertion loss, all of the parameters comprising the insertion loss should be estimated. The relative
contributions of each component to the insertion loss can then be evaluated. Also, the intrinsic properties of the microresonator can be determined more accurately from spectral measurements at the output ports if all of the system transmission efficiencies are known or are accurately estimated.

Input and output coupling efficiency were estimated using a method similar to that reported by He et al. [114] with measured values for a particular device fabricated for this thesis with a weakly-coupled microresonator. This thesis device was one of the test devices used for the electron beam lithography (EBL) process development (EBL_Test3), which had a bus waveguide-microresonator gap of 1,400 nm. A weakly-coupled device was chosen because, for the throughput port of a weakly coupled microresonator, a simplifying assumption of 100% microresonator throughput transmission off-resonance ($T_{\text{res}} = 1$) can potentially be made. Put another way, the effect of the microresonator on the off-resonance optical transmission in the waveguide can be considered to be negligible. The coupling efficiency, $T_{\text{couple}}$, which can be used for the input ($T_{\text{in}}$) or the output ($T_{\text{out}}$), was estimated for the input and the throughput port by the equation

$$T_{\text{couple}} = T_{\text{Fresnel}} \int \int \Psi_{\text{fiber}} \Psi_{\text{wg}} ,$$

(3)

where $T_{\text{Fresnel}}$ is the Fresnel transmission, $\Psi_{\text{fiber}}$ is the fiber electromagnetic field and $\Psi_{\text{wg}}$ is the waveguide electromagnetic field. The Fresnel reflection for normal incidence is given by [115]
\[
R_{\text{Fresnel}} = \left( \frac{n_2 - n_1}{n_2 + n_1} \right)^2,
\]
where \(n_1\) is the refractive index of medium 1 and \(n_2\) is the refractive index of medium 2. 
\(T_{\text{Fresnel}}\) which is defined as \(1-R_{\text{Fresnel}}\) is used to represent the transmitted optical power, which propagates beyond the reflecting interface. The bus waveguides were single mode, so \(\Psi_{\text{wg}}\) would always correspond to either the fundamental TE or TM mode for the bus waveguides. The parameters for a Corning SMF28 single-mode fiber [116] were used to simulate the input field and the parameters for a Corning 62.5/125 multimode fiber [117] were used to simulate the coupling of the output bus waveguide field into the multimode fiber, as these were the fibers used in the experiments.

The overlap integral in (3) between the fiber and the waveguide fields was computed by using the semi-vectorial beam propagation method (BPM) in RSoft software for the TE polarization to generate the fields. Effective refractive indices for each waveguide were also calculated using semi-vectorial BPM for the TE polarization. Fresnel transmissions were calculated by multiplying the Fresnel transmission at the fiber-air interface with the Fresnel transmission at the air-waveguide interface for both ports. To calculate the Fresnel transmission parameter for the output, the effective index of the multimode fiber mode was assumed to be that of the fundamental mode.

Results from TM simulations were found to be close to results from TE simulations, so the TE results were assumed to be a good estimate for both polarizations and only the results for the TE polarization are shown.
Table 3. Power Transmission Parameters for EBL_Test3.

<table>
<thead>
<tr>
<th>System</th>
<th>Power</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P_{in}$</td>
<td>mW</td>
</tr>
<tr>
<td></td>
<td>4.30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Transmission Path Properties</th>
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</thead>
<tbody>
<tr>
<td>cm</td>
</tr>
<tr>
<td>$L_{thru}$</td>
</tr>
<tr>
<td>$L_{drop} - L_{thru}$</td>
</tr>
<tr>
<td>$\alpha_{bus}$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Throughput Path</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transmission Factors</td>
</tr>
<tr>
<td>%</td>
</tr>
<tr>
<td>$T_{ins}$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Input/Output Ports</th>
</tr>
</thead>
<tbody>
<tr>
<td>Input Port</td>
</tr>
<tr>
<td>Effective Refractive Indices (Fundamental TE Mode)</td>
</tr>
<tr>
<td>$n_1$</td>
</tr>
<tr>
<td>$n_2$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Transmission Factors</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
</tr>
<tr>
<td>$T_{Fresnel}$</td>
</tr>
<tr>
<td>$T_{overlap}$</td>
</tr>
<tr>
<td>$T_{couple}$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Throughput Coupler</th>
</tr>
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<tbody>
<tr>
<td>Transmission Factors</td>
</tr>
<tr>
<td>%</td>
</tr>
<tr>
<td>$T_{res}$</td>
</tr>
</tbody>
</table>

¹Difference in propagation path lengths, fixed by design.
²Estimated.
³Estimated from the off-resonance transmission in the throughput spectrum.
⁴Estimated off-resonance throughput coupler transmission assuming symmetric coupling of the microresonator.
The calculated $T_{\text{overlap}}$ for the input (3.34 dB) agrees well with a previous measurement of this parameter (3.5 dB) for a single mode fiber coupled to a 2 µm (height) X 4 µm (width) SU-8 waveguide with an SiO$_2$ substrate and NOA61 ($n=1.54$) cladding [118]. The calculated Fresnel transmissions are quite close to 1, due to the small difference between the effective refractive indices of the fundamental fiber modes and of the fundamental waveguide modes. The total coupling efficiencies are comparable to the measured coupling efficiency of ~32% for a single mode fiber coupled to a 2 µm (height) X 4 µm (width) SU-8 waveguide with an SiO$_2$ substrate and NOA61 ($n=1.54$) upper cladding [118]. The coupling efficiency is also comparable to that measured for a single-mode fiber coupled to an SU-8 waveguide, which had a slightly lower refractive index SU-8 substrate and upper cladding (refractive index contrast of ~0.35%) [109]. For a waveguide height of 4.5 µm and widths of 3, 5, and 10 µm, coupling efficiencies were measured to be ~32%, ~50%, and ~79%, respectively [109].

The simulated input and output coupling efficiencies, along with the measured $T_{\text{ins}}$ for this device and the estimated $T_{\text{res}}$, were used to calculate $T_{\text{prop}}$ with equation (2). For a particular measurement of the transmission spectrum of EBL_Test3, $T_{\text{ins}}$ was measured to be -21.1 dB. Using (2), $T_{\text{prop}}$ was estimated to be -15.0 dB. Using the measured propagation length of 2.21 cm results in an estimated propagation loss of 6.8 dB/cm ($=15.0$ dB/2.21 cm).
2.5 Microresonator Modeling

2.5.1 Transmission

To define $T_{res}$, which is an important part of the insertion loss in microresonators, the transmission spectrum for a microresonator coupled to bus waveguides must be theoretically modeled. The theoretical transmission model is also necessary to calculate the coupling coefficients between the bus waveguides and the microresonator, as well as the microresonator round-trip loss coefficient from measured spectra. The coupling and loss parameters are important, because they are critical factors in determining the Q factor of the device, which is inversely proportional to the LOD.

Theoretically, the microresonator can be coupled to $N$ number of bus waveguides. The two most common coupling configurations, that of one ($N=1$) and two ($N=2$) waveguide coupling, are examined in detail in the following analysis. The trade-offs between the two coupling configurations are briefly examined. The equations describing the important features of the transmission spectra are developed in a specific manner to enable calculation of the coupling and loss coefficients without knowledge of the input and output coupling efficiency in some cases and to generalize the equations to $N$ waveguides in any geometric orientation.

In planar microresonators, light is coupled into and out of the resonator via adjacent bus waveguides as shown in Figure 6.
Figure 6. Waveguide coupling diagram for a vertically-coupled planar microresonator. (a) Single-waveguide coupled. (b) Dual-waveguide coupled.
Light couples into the microresonator and circulates for many cycles before exiting. This physical mechanism produces a series of approximately Lorentzian-shaped spectral features corresponding to the resonant wavelengths of the microresonator. As can be seen in the hypothetical spectra plots of Figure 7, these spectral features appear different, depending on which output port is used for measurement.
Figure 7. Hypothetical transmission spectra for the microresonator device. Important features are labeled and define the notation used in the equations of this section. (a) Throughput port transmission ($T_T$). (b) Drop port transmission ($T_D$).

Periodic nulls corresponding to the resonant wavelengths are observed in the spectrum measured at the throughput port. Periodic peaks are observed in the spectrum measured at the drop port. The spacing between the resonances is called the free spectral range (FSR) and is denoted as $\Delta \lambda_{FSR}$. The width of a spectral feature is usually
defined as the full-width at half-maximum (FWHM) and is denoted as $\Delta \lambda_{\text{FWHM}}$. One important caveat to this definition of the FWHM is that it applies only to resonant features that appear as peaks in the spectrum. The corresponding definition of FWHM for nulls can be derived from the transmission equations for the waveguide-coupled microresonator and is given by equation (20).

The resonant wavelengths ($\lambda_p$) of a microresonator are determined by the resonance condition

$$\beta_p L = 2\pi p,$$  \hspace{1cm} (5)

where $\beta_p$ is the propagation constant of a mode corresponding to the $p$-th resonant wavelength, $L$ is the cavity length, and $p$ is an integer specifying the longitudinal mode and its total phase shift as a multiple of $2\pi$ through one round-trip of the cavity. Using the relationship $\beta_p = 2\pi n_{\text{eff},p}/\lambda_p$, where $n_{\text{eff},p}$ (the $p$ in this usage implies a waveguide mode with particular indices $m$ and $n$) is the effective refractive index for a guided waveguide mode with free space wavelength $\lambda_p$, (5) can be rewritten in terms of the resonant wavelength and the effective index of its corresponding waveguide mode:

$$n_{\text{eff},p} L = \lambda_p p.$$  \hspace{1cm} (6)

The latter form of the resonance condition is useful for relating the change in resonance wavelength to the change in the mode effective index.

The transmission characteristics of a microresonator are important for optimizing insertion loss and Q factor. In the following analysis, analytical expressions of the
microresonator transmission spectrum are derived. These expressions, in combination with (1), can be used to calculate the coupling coefficients between the bus waveguide and the microresonator as well as the internal loss of the microresonator using measured transmission spectra.

By considering the coupling efficiency between the bus waveguides and the microresonator, the transmission characteristics of the resonator can be derived analytically. Yariv showed that using a transfer-matrix method and considering the coupling efficiency between the bus waveguides and the microresonator, the relationships between the microresonator Q factor and the cross-coupling and self-coupling parameters $\kappa$ and $t$ can be derived [119]. The following analysis of single and dual waveguide coupled microresonators is based on Yariv’s approach and utilizes the analysis method published by Cho et al. [120] to derive the transmission expressions. Some of the results of the analysis herein match that of Scheuer et al. [121]. The equations given here differ from those given previously in that the equations can be applied to arbitrary bus waveguide orientations with respect to each other and they represent a complete set of equations describing the important features of the transmission spectra for both the single and the dual waveguide coupled cases in a consistent notation (Yariv’s notation [119]). Such a set of equations was necessary to perform a competent analysis of the measured spectra for the devices fabricated and tested for this thesis as well as a theoretical treatment of the design trade-offs.
Furthermore, this analysis informs the proper measurement of spectra in order to obtain
the most accurate measurements of the coupling and loss parameters.

The transmission analysis makes several assumptions. Coupling is assumed to
be lossless, such that \( \kappa^2 + t^2 = 1 \), the excess phase shift, due to the change in the guided
mode phase constant at the coupling regions, is assumed to be negligible, and gain is not
considered (i.e. all of the coupling and loss parameters must be less than 1).

For the analysis, it is useful to define a parameter, \( Z \), such that the derived
equations are generalized for \( N \) number of coupled bus waveguides. This notation also enables many of the equations to be written in terms of two unknown variables, which simplifies calculations. For the \( N \)-waveguide-coupled case, \( Z \) is defined as

\[
Z = a \prod_{k=1}^{N} t_k ,
\]

where \( a \) is the round-trip loss coefficient, and \( t_k \) is the self-coupling coefficient corresponding to the \( k \)-th bus waveguide. The cavity round-trip loss coefficient, \( a \), is defined as

\[
a = \exp(-\alpha L / 2),
\]

where \( \alpha \) is the exponential loss coefficient for a particular guided mode in the microresonator waveguide. For the cases of single and dual waveguide coupling, the expressions for \( Z \) are given in Table 4.
Table 4. Definitions of Z for 3 types of waveguide-coupled microresonators.

<table>
<thead>
<tr>
<th></th>
<th>Z Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single Waveguide Coupled</td>
<td>$Z = at$</td>
</tr>
<tr>
<td>Dual Waveguide Coupled (asymmetric)</td>
<td>$Z = at_1t_2$</td>
</tr>
<tr>
<td>Dual Waveguide Coupled (symmetric [t_1=t_2])</td>
<td>$Z = at^2$</td>
</tr>
</tbody>
</table>

In this analysis, the exponential loss coefficient $\alpha$ does not include coupling losses to the bus waveguides. These coupling losses are represented by the coupling parameter $t$ for each coupled waveguide. However, it is possible to model the losses due to the out-coupling of light from the microresonator to the bus waveguides as a distributed loss over the length of the cavity if necessary.

2.5.1.1 Single Waveguide Coupled

The simplest case of waveguide coupling is that of a single waveguide coupled microresonator. The single-waveguide coupled case can be treated with a single set of coupled mode equations for the throughput coupler given by [120]

$$
\begin{bmatrix}
    E_{2,1} \\
    E_{4,1}
\end{bmatrix} =
\begin{bmatrix}
    t & j\kappa \\
    j\kappa & t
\end{bmatrix}
\begin{bmatrix}
    E_{1,1} \\
    E_{3,1}
\end{bmatrix}
$$

(12)

and an equation describing the relationship between the intracavity fields, given by [120]

$$
E_{3,1} = a \exp(j\phi)E_{4,1}.
$$

(13)
The $E$ terms in this set of equations are the electric field amplitudes at each part of the coupler, which are defined in Figure 6a, and $\phi$ is the total round-trip phase shift. From this set of equations, the transmission spectrum for the single waveguide coupled case is derived as

$$t_T = \left| \frac{E_{2,1}}{E_{1,1}} \right|^2 = \frac{t^2 + a^2 - 2at \cos(\phi)}{1 + a^2 t^2 - 2at \cos(\phi)} = \frac{1}{t^2} \frac{t^4 + Z^2 - 2t^2 Z \cos(\phi)}{1 + Z^2 - 2Z \cos(\phi)}.$$

(14)

$\phi$ can be given in terms of the quantities of phase constant or effective refractive index using the resonance condition (equations (5) and (6)) of the microresonator,

$$\phi = \beta_{mn}(\lambda)L + \varphi = \frac{2\pi n_{\text{eff},mn}(\lambda)L}{\lambda} + \varphi,$$

(15)

where $\varphi$ is the excess phase shift at the coupler, or in terms of measureable quantities,

$$\phi = 2\pi \frac{(\lambda - \lambda_p)}{\Delta\lambda_{\text{FSR}}} p,$$

(16)

where $\Delta\lambda_{\text{FSR}}$ is the free spectral range, the spacing between the peaks in the spectrum. There is no need to determine $p$, because $\phi$ need only be expressed modulo $2\pi$ for use in the transmission equation. Therefore, (16) can be rewritten as

$$\phi = 2\pi \frac{(\lambda - \lambda_q)}{\Delta\lambda_{\text{FSR}}},$$

(17)

where $\lambda_q$ is a reference resonant wavelength, to calculate $\phi$. This form is useful for fitting the transmission equation models to measured data, because it is based on directly observable quantities in measured spectra. At resonance, when $\phi = 2\pi p$, the transmission at the throughput coupler is derived from (14) as
\[
\left( \frac{t - a}{1 - at} \right)^2 = \frac{1}{\left( t^2 - Z \right)^2} \cdot (1 - Z)^2.
\]

To determine the coupling parameter \( t \) and the cavity loss factor \( a \), an approach that does not require knowledge of the propagation loss or the input/output coupling efficiencies is preferred. Fortunately, quantities that depend only on the cavity coupling and loss parameters are readily obtainable from measured spectral data and these quantities are readily derived from the transmission equation (14). In order to calculate the two unknown parameters \( a \) and \( t \), or equivalently \( Z \) and \( t \) in this case, two equations are necessary. The two equations are those for the observable parameters of extinction ratio and of peak width.

Extinction ratio, which is the ratio of maximum transmission \((\phi = \pi \nu)\) to minimum transmission \((\phi = 2\pi \nu)\) in the vicinity of a resonance spectral feature for the throughput port, is derived from equation (14) as

\[
\left( \frac{t + a}{t - a} \right)^2 \left( \frac{1 - at}{1 + at} \right)^2 = \left( \frac{t^2 + Z}{t^2 - Z} \right)^2 \left( \frac{1 - Z}{1 + Z} \right)^2.
\]

Peak width is the second observable parameter that is needed to determine \( a \) and \( t \). The definition of the FWHM, which is normally measured from a peak in a spectrum, is somewhat unclear when a resonance appears as a null in a spectrum, which is the case when the resonant wavelengths are observed at the throughput port. The width of the null can be utilized to calculate the FWHM directly from the throughput port spectrum. The width of the null in the throughput spectrum at half of maximum is not the same as
the width of the peak at half of maximum. Therefore, to calculate the FWHM directly from the throughput port spectrum, the appropriate transmission level at which to measure the width of the null must be determined. An equation to provide the proper transmission level at which to measure the null width was derived using the transmission equations for the single waveguide coupled case (equation (14)) and for the dual waveguide coupled case (equation (28)):

\[ x_m' = 1 - X (1 - T) \]  

(20)

This equation matches the width of a resonance null (measured at the throughput port) with the width of a resonance peak (measured at the drop port) in the transmission spectrum measured at an arbitrary fraction \( X \) of the maximum transmitted power of the resonance peak. The resonance linewidth, which is inversely proportional to the energy stored in the resonant cavity, an intrinsic property of the microresonator, must be the same from whatever port it is measured. Generally, for measured data that includes waveguide propagation losses and input/output coupling losses, equation (20) cannot be used directly. These factors must be accounted for before (20) can be applied. This property of the throughput port resonances can complicate the accurate measurement of the Q factor from throughput port spectra. However, if the transmission on resonance is very close to zero, which is often the case when high extinction ratio is observed at the throughput port, then (20) can be approximated by (1 – \( X \)) (e.g. half of maximum or 3 dB down from maximum would apply for measurements of the full width at half of
maximum) and applied to spectra measured at the throughput port [65]. For the peak width corresponding to the FWHM, \( X = 0.5 \), the FWHM is derived from equation (14) using equation (20) as

\[
\Delta \lambda_{\text{FWHM}} = \left( \frac{\Delta \lambda_{\text{FSR}}}{\pi} \right) \arccos \left( 2 - \frac{1}{2at} - \frac{at}{2} \right) = \left( \frac{\Delta \lambda_{\text{FSR}}}{\pi} \right) \arccos \left( 2 - \frac{1}{2Z} - \frac{Z}{2} \right). \tag{21}
\]

An expression defining \( Z \) can be derived from (21) and is given by

\[
Z = at = 2 - \left( \cos \left( \frac{\pi}{F} \right) - 4 \cos \left( \frac{\pi}{F} \right) + 3 \right)^{1/2} - \cos \left( \frac{\pi}{F} \right), \tag{22}
\]

where \( F \) is the cavity finesse and is defined as

\[
F = \frac{\Delta \lambda_{\text{FSR}}}{\Delta \lambda_{\text{FWHM}}}. \tag{23}
\]

Thus, \( Z \), the product of \( a \) and \( t \) for the single waveguide coupled case, can determined by using the measured FSR and FWHM with equation (22). A notable detail in the derivation of \( Z \) from (21) is that two solutions exist for \( Z \). Only one of the two solutions ranges between the values of 0 and 1 for all \( F > 0 \), whereas the other solution ranges from 1 to \( \infty \). The former solution is the only valid solution when no gain is present in the system. Another notable detail of this solution for \( Z \) is that it only applies to the peak width at half maximum.

Once \( Z \) is determined, \( a \) and \( t \) must be determined. \( Z \) only provides information about the product of these two factors. The measured extinction ratio provides the additional information necessary to determine \( a \) and \( t \) individually. Equation (19) can be
used to solve for $t$ in terms of the throughput port extinction ratio, $tER$, and the $Z$ parameter. Two solutions result from solving (19) for $t$,

$$
t = \left( \frac{tER^{1/2} + \mp \sqrt{a^2 + t^2 + tER^{1/2} + 1}}{tER^{1/2} + \pm \sqrt{a^2 + t^2 + tER^{1/2} + 1}} \right)^{1/2}
$$

Both solutions are potentially valid, but typically only one of the solutions is physically meaningful. By dividing $Z$ by the two solutions for $t$, two solutions for $a$ will be obtained. Often one of the solutions for $a$ is greater than 1, which implies gain in the resonator. In this case, the correct solution for $a$, and concomitantly $t$, can be identified. For all of the devices measured for this thesis, this method of determining the correct solutions for $a$ and $t$ was applicable.

### 2.5.1.2 Dual Waveguide Coupled

It is often desirable to couple the microresonator to a drop waveguide to measure the spectrum, because the resonances appear as peaks instead of nulls when observed in spectra measured at the drop port. One reason that this would be done is that, because the FWHM is directly measureable from spectra measured at the drop port, the Q factor can be simply and accurately quantified from these spectra. Another reason is that, because the resonances are manifest in the drop port as peaks in the spectrum instead of nulls, there can sometimes be an advantage in signal to noise ratio (SNR) for the power measurements that comprise the spectrum measurement around the resonant wavelengths, depending on the measurement instrumentation and the strength of the
coupling between the microresonator and the bus waveguides. All of the sensors that were fabricated and tested for this thesis were dual waveguide coupled microresonators.

For a dual waveguide coupled structure, three sets of equations, two defining the relationships between the fields at each coupler and one defining the relationships between the intracavity fields, are necessary to define the transmission spectrum. The field relationships for the throughput coupler, the drop coupler, and the intracavity fields are

\[
\begin{bmatrix}
E_{2,1} \\
E_{4,1}
\end{bmatrix} =
\begin{bmatrix}
t_1 & j\kappa_1 \\
 j\kappa_1 & t_1
\end{bmatrix}
\begin{bmatrix}
E_{1,1} \\
E_{3,1}
\end{bmatrix},
\]

(25)

\[
\begin{bmatrix}
E_{2,2} \\
E_{4,2}
\end{bmatrix} =
\begin{bmatrix}
t_2 & j\kappa_2 \\
 j\kappa_2 & t_2
\end{bmatrix}
\begin{bmatrix}
E_{1,2} \\
E_{3,2}
\end{bmatrix},
\]

(26)

\[
\begin{bmatrix}
E_{3,1} \\
E_{3,2}
\end{bmatrix} =
\begin{bmatrix}
0 & a^{1-FD} \exp[j(1-FD)\phi] \\
 a^{FD} \exp[j(FD)\phi] & 0
\end{bmatrix}
\begin{bmatrix}
E_{4,1} \\
E_{4,2}
\end{bmatrix},
\]

(27)

respectively. \(FD\) is the fractional distance of the drop coupler from the throughput coupler, which is the distance measured in the propagation direction between the throughput port and the drop port divided by the total cavity length. For example, for the dual waveguide coupled microresonator depicted in Figure 6b, \(FD=0.5\). The drop port transmission is derived from this set of equations as

\[
\begin{align*}
dT &= \frac{E_{2,2}^2}{E_{1,1}} \\
&= \frac{a^{2FD}\kappa_1^2\kappa_2^2}{1 + a^2 t_1^2 t_2^2 - 2at_1 t_2 \cos(\phi)} \cdot \left( \frac{Z}{t_1 t_2} \right)^{2FD} \cdot \frac{(1-t_1^2)(1-t_2^2)}{1 + Z^2 - 2Z \cos(\phi)}
\end{align*}
\]

(28)
where $t_1$ and $t_2$ and $\kappa_1$ and $\kappa_2$ are the coupling parameters for couplers 1 and 2, respectively. Usually, the throughput coupler is identified as 1 and the drop coupler is identified as 2.

At the drop port, the equations relevant to measurable features in the spectrum, derived from equation (28), are

$$d_T = \frac{a^{2\text{FD}} \kappa_1^2 \kappa_2^2}{(at_1t_2 - 1)^2} \left( \frac{Z}{t_1t_2} \right)^{2\text{FD}} \frac{(1 - t_1^2)(1 - t_2^2)}{(Z - 1)^2},$$

(29)

$$d_{ER} = \frac{(at_1t_2 + 1)^2}{(at_1t_2 - 1)^2} = \frac{(Z + 1)^2}{(Z - 1)^2},$$

(30)

$$\Delta\lambda_{\text{FWHM}} = \left( \frac{\Delta\lambda_{\text{FSR}}}{\pi} \right) \arccos \left( 2 - \frac{1}{2at_1t_2} - \frac{at_1t_2}{2} \right) = \left( \frac{\Delta\lambda_{\text{FSR}}}{\pi} \right) \arccos \left( 2 - \frac{1}{2Z} - \frac{Z}{2} \right).$$

(31)

Note that the equation for FWHM (31) is necessarily the same as that for the single waveguide coupled case (21), which means that the solution for $Z$ derived from that equation will be the same as for the single waveguide coupled case as well. The solution for $Z$ is found from solving (30) or (31) for $Z$ and is given by

$$Z = at_1t_2 = \frac{d_{ER}^{1/2}}{d_{ER}^{1/2} + 1}$$

or

$$Z = at_1t_2 = 2 - \left( \cos^2 \left( \frac{\pi}{F} \right) - 4 \cos \left( \frac{\pi}{F} \right) + 3 \right)^{1/2} - \cos \left( \frac{\pi}{F} \right),$$

(32)

(33)

respectively. Because the Q factor and FSR are intrinsic properties of the waveguide-coupled microresonator, by extension the FWHM, $F$, and $Z$, which are related to these parameters, are also intrinsic to the waveguide-coupled microresonator. Therefore,
these parameters will always be the same, regardless of the bus waveguide output port from which they are measured. Also notable is the fact that only $Z$ can be determined from the drop port spectrum, because none of the parameters $a$, $t_1$, or $t_2$, appear independently in the extinction ratio equation (30). Therefore, to obtain solutions for these parameters, the spectrum at the throughput port must be measured. Solving the system of equations for the throughput port transmission spectrum yields the same equations in terms of $Z$ and $t$ as for the single waveguide coupled case, except that $t_1$ is substituted for $t$ and $Z$ is defined as $at_1t_2$. The relevant equations for the throughput port for the dual waveguide coupled case are

\[
T' = \frac{t_1^2 + a^2t_2^2 - 2at_1t_2 \cos(\phi)}{1 + a^2t_1^2t_2^2 - 2at_1t_2 \cos(\phi)} = \frac{1}{t_1^2} \left( t_1^4 + Z^2 - 2t_1^2Z \cos(\phi) \right), \tag{34}
\]

\[
T' = \frac{(t_1 - at_2)^2}{(at_1t_2 - 1)^2} = \frac{1}{t_1^2} \left( t_1^4 - Z \right)^2, \tag{35}
\]

\[
ER' = \frac{(t_1 + at_2)^2}{(t_1 - at_2)^2} \frac{(at_1t_2 - 1)^2}{(at_1t_2 + 1)^2} = \frac{(t_1^2 + Z)^2 (Z - 1)^2}{(t_1^2 - Z)^2 (Z + 1)^2}. \tag{36}
\]

The solutions for $t_1$ are found from solving for $t_1$ from equation (36) in terms of $Z$ and $ER$:
\[
t_1 = \left( \frac{t'ER^{1/2} a t_1 t_2 \mp at_1 t_2 \pm a^2 t_1^2 t_2^2 + t'ER^{1/2} a^2 t_1^2 t_2^2}{t'ER^{1/2} a t_1 t_2 \mp at_1 t_2 + t'ER^{1/2} \pm 1} \right)^{1/2} = \\
\left( \frac{t'ER^{1/2} \mp Z \pm Z^2 + t'ER^{1/2} Z^2}{t'ER^{1/2} \mp Z + t'ER^{1/2} \pm 1} \right)^{1/2}.
\]

(37)

The solutions for the coupling parameter at the throughput coupler for the dual waveguide case exactly match that for the single waveguide case with a different definition of \(Z\), because there are now two waveguide outputs in the system. Because \(t_1\) can always be calculated in terms of \(t'ER\) and \(Z\) for any number of bus waveguides from the spectrum measured at the throughput port, the throughput port spectrum is a critical measurement for the characterization of the microresonator transmission and loss parameters with \(N\) number of coupled bus waveguides. Furthermore, because the Q factor and the FSR of the microresonator are intrinsic properties of the microresonator, which implies that the FWHM and the \(Z\) are also intrinsic properties, the \(Z\) parameter can be calculated from a spectral measurement at any port. Because it is not necessary to utilize equation (20) to measure the FWHM from the spectrum measured at the drop port, the calculation of the \(Z\) parameter for a dual waveguide coupled microresonator from the drop port spectrum is preferable. For the generalized equations describing the \(N\)-waveguide-coupled case, see Appendix A: Transmission Analysis for an \(N\)-Waveguide Coupled Microresonator.
2.5.1.3 Measurement Methods for Dual Waveguide-Coupled Systems

To be able to apply the theory to measured data, it is important that measurement methods be informed by the requirements of the theoretical models. For the dual waveguide coupled case, there are three measurement techniques that can be utilized to measure the round-trip loss coefficient $a$, and the coupling parameters $t_1$ and $t_2$. These methods were formulated by an evaluation of the transmission equations for the dual waveguide coupled microresonator. In the following discussion, three methods are described and their advantages and disadvantages are evaluated.

2.5.1.3.1 Throughput Spectrum Measurement with the Symmetric Coupling Assumption

Symmetric coupling is a good assumption when the couplers are geometrically symmetric and when the phase constants of the microresonator waveguide and the bus waveguide optical modes are expected to be matched. It is also a good assumption in the highly decoupled microresonator case, where $t_1\approx t_2\approx 1$. If symmetric coupling can be assumed, only the throughput port spectrum needs to be measured to determine the model parameters. This assumption was used previously to extract the microresonator round-trip loss coefficient $a$ from measured spectra [122]. The major advantages with this method are that it requires only one spectral measurement to be made at the throughput port and it does not require knowledge of the input/output coupling efficiencies or the bus waveguide propagation loss. The drawback is that it will not reveal any information about the coupling asymmetry in the system.
2.5.1.3.2 Spectral Measurement using the Drop Waveguide as the Throughput Waveguide

Deviations from symmetric coupling often occur when phase mismatch or misalignment between the microresonator and the bus waveguides is present due to fabrication errors or by design. By reversing the measurement, such that the drop waveguide is used as the input waveguide, \( t_2 \) can be calculated from the measured spectrum using (37). This method also does not require knowledge of the input/output coupling efficiencies or the bus waveguide propagation loss. The drawback of this method is that it will not work in the case that phase mismatch is a significant factor in the coupling asymmetry. This phenomenon is explained by the coupled mode theory, which predicts that the coupling efficiency of a directional coupler with a specific length will not be the same from waveguide \( a \) to \( b \) as it is from waveguide \( b \) to \( a \), unless the coupling modes in each waveguide have equal phase constants or are orthogonal to each other [115]. For example, if both couplers were geometrically the same, but the waveguides were phase mismatched, then the measurement performed in this manner would yield the same value for \( t_2 \) as for \( t_1 \), providing no information about the coupling asymmetry. Furthermore, the devices that were tested for this thesis could not be measured in this manner, because light propagating in the drop waveguide could only be measured in one direction (see Figure 1 for the optical system diagram).
2.5.1.3.3 Ratio of Throughput Maximum Transmission to Drop Maximum Transmission

This method, like the previously described method, enables characterization of asymmetric structures. It is also resilient to coupling asymmetry induced by phase mismatch and provides a means to calculate $a$, $t_1$, and $t_2$ without using the throughput as the input waveguide. This method was necessary to calculate parameters without the assumption of symmetric coupling for the devices measured in this thesis, because the throughput waveguide could not be utilized as the input waveguide. In order to utilize this method, a measurement of both the throughput port spectrum and the drop port spectrum is needed. The coupling parameter $t_2$ is calculated from the ratio of the maximum power at the drop port to the maximum power at the throughput port. Generally, measurements or estimates of the input coupling efficiency, the output coupling efficiency, and the bus waveguide propagation loss are also required. However, knowledge of the input coupling efficiency is typically not required as long as it is held constant for both spectrum measurements.

The maximum power at the drop port corresponds to the power on-resonance given by equation (35). The maximum power at the throughput port corresponds to the power off-resonance, in which case the round trip phase change $\phi$ is a multiple of $\pi$. The off-resonance ($\phi = \pi p$) transmission for the throughput port is derived from equation (34) and is given by
\[
\left(\frac{t_1 + at_2}{at_1 t_2 + 1}\right)^2 = \frac{1}{t_1^2} \frac{(t_1^2 + Z)^2}{(Z + 1)^2}.
\] (38)

The ratio of the drop port transmission on-resonance to the throughput port transmission off-resonance is determined by dividing equation (29) by equation (38) and is given by

\[
\frac{d}{n}T = \frac{a^2 F D \kappa_1^2 \kappa_2^2 (at_1 t_2 + 1)^2}{(at_1 t_2 - 1)^2 (t_1 + at_2)^2} = \left(\frac{Z}{t_1 t_2}\right)^{2 F D} \frac{(1 - t_1^2)(1 - t_2^2)}{(Z - 1)^2 (t_1^2 + Z)^2}.
\] (39)

Rearranging this equation yields a solvable equation for \( t_2 \) in terms of \( t_1 \):

\[
(1 - t_2^2) \left(\frac{1}{t_2}\right)^{2 F D} = \frac{d}{n}T \left(\frac{Z}{t_1}\right)^{2 F D} \frac{(Z - 1)^2 (t_1^2 + Z)^2}{t_1^2 (1 - t_1^2)(Z + 1)^2}.
\] (40)

To determine the ratio of the drop port transmission on-resonance to the throughput port off-resonance from the measured power, \( d_P \) and \( n_P \), which denote the measured power at the drop port on-resonance and the measured power at the throughput port off-resonance, respectively, the insertion loss, defined by equations (1) and (2), must be considered. Expanding equation (1) and the insertion loss in equation (1), which is given by equation (2), in terms of the parameters for the on-resonance transmission at the drop port and the off-resonance transmission at the throughput port, two equations are obtained that relate the output power for each port to the input power:

\[
d_P = T_{in} T_{prop, drop} \left(\frac{d_T}{n_T}\right) P_{in}, \text{ and}
\] (41)

\[
n_P = T_{in} T_{prop, thru} \left(\frac{d_T}{n_T}\right) P_{in}.
\] (42)

Dividing equation (41) by equation (42) and rearranging yields the equation
\[
\frac{dT}{dT_{\text{aq}}} = \frac{dP}{dP_{\text{in}}} \frac{T_{\text{in}}}{T_{\text{prop,thru}}} \frac{T_{\text{out,thru}}}{T_{\text{out,drop}}} \approx \frac{dP}{dP_{\text{in}}} P_{\text{out,drop}} \exp\left[\alpha_{\text{bus}} \left(L_{\text{drop}} - L_{\text{thru}}\right)\right],
\]  

(43)

where \(\alpha_{\text{bus}}\) is the propagation loss in the bus waveguide, \(L_{\text{drop}}\) is the propagation path length corresponding to the drop port, and \(L_{\text{thru}}\) is the propagation path length corresponding to the throughput port. The approximation in equation (43) of equal output coupling efficiencies at the drop port and the throughput port is reasonable if the fiber coupling efficiency at each output port is maximized by optimal alignment of the fiber with the waveguide. The approximation of equal output coupling efficiency might also be improved by polishing the waveguide output facets and by using automated fiber alignment. Further simplification of equation (43) is possible if the optical system is designed such that \(L_{\text{drop}} = L_{\text{thru}}\).

The advantage of this method is the ability to accurately quantify all of the coupling parameters, \(a\), \(t_1\), and \(t_2\), for the general case of asymmetric coupling. The disadvantage is that the accuracy of this method is affected by the accuracy with which the output coupling efficiencies are known (or the error in the approximation of equal output coupling efficiencies) and the accuracy of the propagation loss estimate.

### 2.5.2 Quality Factor

Quality factor (Q), which is defined as the ratio of energy stored to energy lost per round trip in a resonant cavity, is a figure of merit to describe the performance of a resonant cavity. The Q factor is also relevant to the performance of a resonant cavity sensor, because the limit of detection for resonant wavelength shifts is inversely
proportional to Q for an intensity noise limited wavelength resolution [65]. The total resonator Q factor is limited by several loss producing mechanisms, each with its own exponential loss coefficient \( \alpha \). It is possible to calculate a limiting Q factor associated with each source of optical loss. By summing the inverses of all of the Q factors calculated for each optical loss source and taking the inverse of the total, the total cavity Q factor can be calculated. The general form of that equation is written as [123]

\[
\frac{1}{Q} = \sum_i \frac{1}{Q_i}.
\] (44)

The primary losses encountered are absorption and Rayleigh scattering by the material \( Q_{\text{mat}} \), surface scattering \( Q_{\text{ss}} \), caused by coupling of light into non-functional guided modes, leaky modes, and radiation modes of the waveguide facilitated by the rough surfaces, bending loss \( Q_{\text{bend}} \), caused by leakage of light caused by the waveguide curvature, loss to the coupling waveguides \( Q_{\text{coup}} \), caused by the coupling between the bus waveguides, and substrate loss \( Q_{\text{sub}} \), caused by evanescent coupling of the guided waves into a substrate material with a higher refractive index than the waveguide core material [122, 124, 125]. Therefore, total Q factor for a microresonator can be estimated as

\[
Q^{-1} = Q_{\text{mat}}^{-1} + Q_{\text{ss}}^{-1} + Q_{\text{bend}}^{-1} + Q_{\text{coup}}^{-1} + Q_{\text{sub}}^{-1}.
\] (45)

The corresponding exponential loss coefficients are, in the same order, \( \alpha_{\text{mat}}, \alpha_{\text{ss}}, \alpha_{\text{bend}}, \alpha_{\text{coup}}, \) and \( \alpha_{\text{sub}} \). The total exponential loss coefficient, \( \alpha \), is given by

70
\[ \alpha = \alpha_{\text{mat}} + \alpha_{\text{ss}} + \alpha_{\text{bend}} + \alpha_{\text{coup}} + \alpha_{\text{sub}}. \]  \hfill (46)

Each loss mechanism can be mitigated by proper design. The material absorption loss can be minimized by choosing an interrogation wavelength for which the material has low loss, or by choosing a material with low absorption loss at a particular wavelength. The surface scattering loss can be minimized by ensuring that smooth surfaces are formed in the fabrication process. Bending loss is decreased by keeping bend radii large compared to the optical wavelength. Coupling loss is decreased by decoupling the microresonator from its bus waveguides, which is usually achieved by increasing the distance between the microresonator and any coupling waveguides. Coupling can also be reduced by mismatching the propagation constants of the bus waveguide and the microresonator waveguide, or by decreasing the coupler length. Substrate loss occurs when a substrate contains a layer of material with a refractive index equal to or greater than the waveguide core material’s refractive index to which the guided mode evanescently couples. Substrate loss is reduced typically by increasing the thickness of a low refractive index buffer layer, such as SiO₂, between the waveguide and the high refractive index substrate, such as Si.

The total quality factor for a waveguide coupled microresonator can be measured from its spectrum and is given by [122]

\[ Q = \frac{\lambda_p}{\Delta \lambda_{\text{FWHM}}}. \]  \hfill (47)
The FWHM can also be calculated using (31) with the measured free spectral range (FSR). The FSR is derived from the resonance condition and is given by [122, 126]

\[ \Delta \lambda_{FSR} = \frac{\lambda_p^2}{n_{g,p} L} \] (48)

where \( n_g \) is the group refractive index, the ratio of the speed of light to the velocity of a wave packet (a signal which consists of a range of wavelength components) in the waveguide, the group velocity, which is defined as [122]

\[ n_{g,p} = n_{eff,p} - \lambda_p \frac{dn_{eff,p}}{d\lambda}. \] (49)

Using equation (48) and equation (31) in equation (47), the Q factor for a waveguide coupled microresonator is [121]

\[ Q = \frac{\lambda_p}{\Delta \lambda_{FWHM}} = \frac{L m_{g,p}}{\lambda_p} \arccos \left( 2 - \frac{1}{2Z} - \frac{Z}{2} \right)^{-1}. \] (50)

By first expanding the cosine term in the transmission equation in a 2nd order Taylor series and then solving for the full-width half maximum, the total Q factor can be derived in a simpler approximate form as [122, 126]

\[ Q = \frac{\lambda_p}{\Delta \lambda_{FWHM}} \approx \frac{L m_{g,p}}{\lambda_p} \frac{\sqrt{Z}}{1 - Z}. \] (51)

This equation is more broadly applicable than (50), because the domain of Z is not restricted to values of Z for which the FWHM is defined in the transmission spectrum. Often, \( n_g \) and/or \( n_{eff} \) are approximated as being equal to the microresonator material’s refractive index in order to estimate the Q factor without solving for the microresonator
waveguide dispersion equation. A useful approximation of (51) can be made to calculate the intrinsic (uncoupled) cavity Q factor for a low loss cavity \(a \approx 1\) by setting \(t = 1\), using the approximation \(1 - a \approx \alpha L/2\) in the denominator, and \(a \approx 1\) in the numerator [122, 123]:

\[
Q_{\text{int}} = \left(\frac{1}{Q_{\text{mat}}} + \frac{1}{Q_{\text{bend}}} + \frac{1}{Q_{\text{ss}}} + \frac{1}{Q_{\text{sub}}}\right)^{-1} \approx \frac{2\pi n_{g,p}}{\alpha \lambda_p}.
\] (52)

This equation will be used later in the definition of a simple figure-of-merit (FOM) for microresonators that is inversely proportional to the exponential loss coefficient.

In the following section, a method for theoretically calculating the sensitivity is described. The sensitivity model was important for this work, because it helped to identify what variables could be adjusted to optimize the sensitivity.

2.5.3 Sensitivity

To determine the sensitivity of a microresonator to changes in the cladding refractive index (e.g. a solution in contact with the microresonator), the relationship between the waveguide dispersion and the cavity resonance condition should be accurately defined. For a waveguide coupled microresonator, the resonance condition, considering the resonance condition (equation (5)) and the dependence of the phase constant on the refractive index in the sensing region \(n_s\) and on the resonant wavelength \(\lambda_p\), is given by

\[
\beta_{mn}(n_s, \lambda_p) L = 2\pi p.
\] (53)
From (53), a variety of simple and useful relationships between the sensitivity of the microresonator and the solutions to its dispersion equation, $\beta_{mn}$, can be derived. According to equation (53), a change in the resonant wavelength is generally necessary to satisfy the resonance condition when a change in $n_s$ occurs for a specific longitudinal mode $p$. (53) by itself can be used to rigorously solve for the change in resonant wavelength given a change in $n_s$ with a root-finding algorithm and a mode solver, because the ratio $2\pi p/L$, and thus the propagation constant, $\beta$, for a particular longitudinal resonance, $p$, remains constant with changes in $n_s$. By replacing $n_s$ with $n_{ci}$, which is the refractive index for the entire region above the substrate outside of the waveguide core (the cladding), simple waveguide models, such as the slab waveguide model for planar waveguides with effectively one-dimensional structure, or the effective index method (EIM) for channel waveguides with two-dimensional structure (see Figure 5) [115, 127], can be used to calculate $\beta$ for different values of $\lambda$. The sensitivity defined in this manner is typically referred to as the bulk sensitivity and can be used to compare the sensitivities of different microresonators.

Using equation (53) along with the slab waveguide formula and the effective index method (EIM) to solve for $n_{eff}$, curves of sensitivity versus height were calculated for five different waveguide widths for the TE$_{00}$ mode of an SU-8 channel waveguide on a SiO$_2$ substrate immersed in water. The EIM models a rectangular channel waveguide as two slab waveguides oriented perpendicularly to each other and superimposed. This
model is generally accurate for waveguides in which waveguiding is much more strong in the y (vertical) direction than in the x (horizontal) direction [115]. Usually, this condition is met only when the waveguide width is greater than its height [115]. The refractive index perturbation was applied to the entire cladding and was $1 \times 10^{-3}$. The resulting wavelength perturbation was determined by iteratively solving equation (53) for the resonant wavelength with the perturbed cladding refractive index. Sensitivity for a particular waveguide width and height was estimated to be the difference between the resonant wavelength calculated after the index perturbation and the starting resonant wavelength ($\delta \lambda_p$) divided by the refractive index perturbation in the sensing region ($\delta n_s$), which was defined to be the entire cladding region. This method for estimating sensitivity will hereafter be referred to as the iterative method. The estimated sensitivity for the TE$_{00}$ mode for a range of waveguide heights and several widths is shown in Figure 8.
Figure 8. Sensitivity of the TE₀₀ modes versus waveguide height and width for an SU-8 waveguide on a SiO₂ substrate. Lighter shades of blue correspond to the waveguide widths ∞ (slab waveguide), 4 μm, 2 μm, 1.5 μm, and 1 μm. The dashed black line indicates roughly the region of best accuracy (width>height) for the EIM.

The curves show that sensitivity increases as waveguide width and height decrease, up to a point near cut-off. The curves also indicate that minimizing the waveguide width would produce the most significant gains in sensitivity for this waveguide structure refractive index profile. A lower limit to the reduction in the width and height of a waveguide is usually set by the cut-off of the fundamental mode, which is the point at which the effective index of the TE₀₀ mode becomes equal to the refractive index of the lowest cladding material surrounding the waveguide core, which is 1.444 (SiO₂) for this waveguide structure refractive index profile. Modes of the waveguide with effective refractive indices below cut-off, which are called leaky modes, have high propagation losses. The high propagation loss of leaky modes usually excludes them from practical use in optical devices. For bent waveguides, as in the case of the microresonator
waveguide, the lower limit in the waveguide width and height can be significantly greater than that set by the mode cut-off, because propagation losses caused by the waveguide curvature can become prohibitively large well before cut-off.

While the sensitivity curves generated with the iterative method indicate the trend in the sensitivity, the waveguide dimensions corresponding to the highest sensitivity waveguides do not meet the accuracy conditions of the EIM. To more accurately model the sensitivity model the waveguides with dimensions that optimize sensitivity, numerical methods must be employed to accurately calculate the phase constants. Because numerical simulations are much more time consuming than the EIM method, it was necessary to define a small space of waveguide widths and heights to simulate.

The resonance condition is often written in terms of the effective refractive index, which provides a more intuitive description of the guided mode. The effective refractive index of modes of the waveguide can be readily compared to the cladding refractive index to determine if the mode is above cut-off and also directly indicates the phase velocity of the mode with respect to the speed of light. Rewriting equation (53) in terms of the effective refractive index gives

\[ \frac{d}{dx} n_{\text{eff,mod}}(n_s, \lambda_p) = p \lambda_p \cdot \frac{L}{\lambda} \quad (54) \]

To reduce the time required to estimate the sensitivity using equation (54), an approximation for the sensitivity explicitly in terms of the mode effective refractive
indices for three different conditions can be derived from (53). An equation for the sensitivity can be determined by estimating the shift in the wavelength necessary to maintain a fixed phase constant through a perturbation of the refractive index, a condition which is implied by the resonance condition, in the sensing region. This approach involves approximating the variation of the phase constant with respect to \( \lambda_p \) and \( n_s \) with a first order derivative by taking the first order Taylor expansion of the phase constant with respect to \( \lambda_p \) and \( n_s \) and then solving for \( d\lambda_p/dn_s \). The following expression for sensitivity is obtained from this approach [126, 128-130]:

\[
S = \frac{d\lambda_p}{dn_s} = \frac{\lambda_p}{n_{g,nn}(n_s, \lambda_p)} \frac{\partial n_{\text{eff},mn}(n_s, \lambda_p)}{\partial n_s},
\]

(55)

where \( n_{g,nn} \) is the group effective refractive index, given by [122]

\[
n_{g,nn} = n_{\text{eff},mn} - \lambda_p \frac{\partial n_{\text{eff},mn}}{\partial \lambda_p}.
\]

(56)

The inclusion of the group index term is sensible, because it accounts for the waveguide dispersion, much in the same manner as the inclusion of the group index term in the expression for the free spectral range. Previous work [128, 129] showed excellent agreement between measured sensitivity and predicted sensitivity by estimating the derivatives of (55) and (56) with first order finite differences.

In order to utilize equations (55) and (56), the derivatives must be estimated with finite differences. A first order difference provides the most efficient estimate of the derivative, because it utilizes the smallest number of terms. By using a first order finite
difference to estimate the derivatives, the derivatives in equations (55) and (56) can be estimated by as few as three mode effective refractive indices, which can be accurately calculated by using numerical methods. By using the iterative method to calculate sensitivity for a waveguide with a particular refractive index profile, an optimal first order finite difference estimate for the differential equations for $S$ and $n_s$ was determined. The optimal condition was minimization of the relative error of solutions to equation (55) with respect to the solutions using the iterative method for a small, positive perturbation in the waveguide cladding refractive index ($1 \times 10^{-3}$ RIU) and a small, positive perturbation in the resonant wavelength (0.1 nm) for a particular waveguide structure. These perturbations are relevant for the most common sensing scenario in which there is a net increase in refractive index, due to an increase in mass density, in the sensing region. The analysis led to the following discretization for the sensitivity equations:

$$S = \frac{\lambda_{p,f} - \lambda_{p,i}}{n_{s,f} - n_{s,i}} = \frac{\Delta \lambda_p}{\Delta n_s} = \frac{\lambda_{p,i}}{n_{g,mm}} \frac{n_{\text{eff,mm}}(\lambda_{p,i}, n_s + \delta n_s) - n_{\text{eff,mm}}(\lambda_{p,i}, n_s)}{\delta n_s}$$

(57)

$$n_{g,mm} = n_{\text{eff,mm}}(\lambda_{p,i}, n_s + \delta n_s) - \lambda_{p,i} \frac{n_{\text{eff,mm}}(\lambda_{p,i} + \delta \lambda_p, n_s + \delta n_s) - n_{\text{eff,mm}}(\lambda_{p,i}, n_s + \delta n_s)}{\delta \lambda_p},$$

(58)

where $\delta n_s$ is the refractive index perturbation in the sensing region, $\delta \lambda_p$ is the perturbation applied to the resonant wavelength to determine the group refractive index, and $\lambda_{p,i}$ and $\lambda_{p,f}$ are the initial and the final resonant wavelengths, respectively. This discretization scheme is similar to, but slightly different from that used previously.
[130], in that the group refractive index is estimated for the perturbed refractive index condition, rather than the initial refractive index condition. Only three waveguide simulations to obtain phase constants for three different conditions are needed to accurately solve for the sensitivity for a particular waveguide using equations (57) and (58). This method for estimating the waveguide sensitivity will hereafter be referred to as the explicit method, because the sensitivity can be determined explicitly without iterations with this method. Using the variational theory of waveguides (see Appendix B: Application of the Variational Theory to Microresonator Sensors), the relationship between the field overlap factor and sensitivity is derived as [65]

\[
S \approx \frac{\lambda_{p,i}}{n_{g,p,i}} \Gamma_s,
\]

(59)

where \( \Gamma_s \) is the fraction of the total field power present in the sensing region.

For design purposes, equation (59) is conceptually useful, because it shows that increasing the fraction of the field intensity in the sensing region improves the sensitivity of the sensor. It should be noted that this approximation is exact only for the case of a slab waveguide with \( n_s = n_{\text{eff}} \) [131], and, therefore, provides at best only an approximation of the sensitivity for channel waveguides with \( n_s \neq n_{\text{eff}} \).

Some general conclusions about waveguide sensing can be made by examining equation (59). According to equation (59), the theoretical maximum sensitivity for a waveguide in the limit of a waveguide of zero size (\( \Gamma_s = 1 \)) and assuming the group velocity cannot exceed the velocity of light in vacuum (\( n_{g,p,i} = 1 \)) is equal in magnitude to
the wavelength and is given in units of m/RIU (typically nm/RIU). More generally, this sensitivity limit is a theoretical limitation on waveguide sensors that utilize a single mode for sensing [132]; that is, for waveguide sensors in which the phase constant of the monitored mode is the same before and after a perturbation of the refractive index profile of the waveguide. The resonance condition (equation (5)) for a microresonator implies a fixed phase constant for a particular longitudinal mode \( p \) through perturbations in the cladding refractive index. Therefore, the microresonator is a single-mode waveguide sensor and is subject to this limitation. Theoretically, this limit can be exceeded if the microresonator were made with a material exhibiting anomalous dispersion or resonant behavior near the probe wavelength, in which case the group index can be less than one [133].

2.5.4 Figure of Merit and Limit of Detection

Using the theoretical methods previously described, the Q factor and sensitivity can be estimated from basic waveguide parameters. Using these two factors, the figure of merit (FOM), which is inversely proportional to the limit-of-detection (LOD), can be calculated. Using the equations (52) and (59) for \( Q \) and \( S \), respectively, the following intrinsic FOM, the FOM for an uncoupled microresonator, is defined as [65]

\[
FOM_{\text{int}} = Q_{\text{int}} * S \approx \frac{2\pi}{\alpha} \Gamma_s, \tag{60}
\]

where the approximation is due to the approximation made for equation (59). This FOM was derived by Hu et al. [65] for a wavelength interrogated device. For a
microresonator coupled to bus waveguides, the coupling loss to the bus waveguides can be modeled as a distributed loss over the length of the microresonator, $\alpha_{coup}$, and included in the total exponential loss coefficient, $\alpha$. In the case that $\alpha_{coup}$ is included in $\alpha$, the FOM is given by

$$FOM = Q \cdot S \approx \frac{2\pi}{\alpha} \Gamma_s,$$  \hspace{1cm} (61)

where $Q$ is the quality factor measured for a waveguide coupled microresonator and FOM is the figure of merit for a waveguide coupled microresonator. According to equation (61), a microresonator sensor is improved by reducing its propagation loss, which is comprised of the material, bending, surface scattering, and coupling losses, and by increasing the field overlap of the guided mode propagating in the microresonator waveguide with the sensing region. Hu et al. showed, using Monte Carlo methods, that $1/(Q \cdot S)$ was proportional to the microresonator sensor’s LOD [65]. Thus, the limit of detection for a waveguide coupled microresonator can be written as

$$LOD \propto \frac{1}{FOM} = \frac{1}{QS}.$$ \hspace{1cm} (62)

This FOM is useful for comparing the performance of different microresonator sensors. Table 5 lists many of the planar microresonators reported to date with relevant properties and FOMs.
Table 5. Sensitivities, Q factors, and other relevant parameters for a variety of planar microring resonators. Results for devices fabricated and tested by this author are shown in bold.

<table>
<thead>
<tr>
<th>Material (core/substrate)</th>
<th>Refractive Index (core/substrate)</th>
<th>Diam. (μm)</th>
<th>w,d (μm)</th>
<th>λ (nm)</th>
<th>Q</th>
<th>S (nm/RIU)</th>
<th>FOM (10^6 nm/RIU)</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si/SiO₂</td>
<td>3.43/1.44</td>
<td>~40</td>
<td>0.5, 0.2</td>
<td>1550</td>
<td>43,000 ⁶</td>
<td>163</td>
<td>7</td>
<td>[94]</td>
</tr>
<tr>
<td>Chalcogenide glass</td>
<td>2.06/1.44</td>
<td>40</td>
<td>0.5, 0.45</td>
<td>1550</td>
<td>20,000 ⁶</td>
<td>182</td>
<td>3.7</td>
<td>[88]</td>
</tr>
<tr>
<td>SU-8/SiO₂</td>
<td>1.57/1.44</td>
<td>2000</td>
<td>2, 2</td>
<td>1310</td>
<td>33,000 ⁶</td>
<td>70</td>
<td>2.3</td>
<td>[106]</td>
</tr>
<tr>
<td>ZPU13-430/LFR-S708U (Polymers)</td>
<td>1.43/1.375</td>
<td>800</td>
<td>2.8, 2</td>
<td>1550</td>
<td>10,000 ⁶</td>
<td>200</td>
<td>2</td>
<td>[87]</td>
</tr>
<tr>
<td>Chalcogenide glass/SiO₂</td>
<td>1.785/1.44</td>
<td>120</td>
<td>Not given</td>
<td>1550</td>
<td>12,000 ³</td>
<td>141</td>
<td>1.7</td>
<td>[66]</td>
</tr>
<tr>
<td>SU-8/SiO₂</td>
<td>1.57/1.44</td>
<td>500</td>
<td>1.5, 1.9</td>
<td>1550</td>
<td>15,000 ³</td>
<td>82</td>
<td>1.2</td>
<td>[134]</td>
</tr>
<tr>
<td>Polystyrene/SiO₂</td>
<td>1.57/1.44</td>
<td>90</td>
<td>2.3, 1.85</td>
<td>1550</td>
<td>20,000 ³</td>
<td>50</td>
<td>1</td>
<td>[105]</td>
</tr>
<tr>
<td>SU-8/SiO₂</td>
<td>1.57/1.44</td>
<td>500</td>
<td>1.2, 2.0</td>
<td>1550</td>
<td>8,400 ³</td>
<td>89</td>
<td>0.75</td>
<td>[134]</td>
</tr>
<tr>
<td>Si/SiO₂</td>
<td>3.43/1.44</td>
<td>10 (3⁴)</td>
<td>0.5, 0.22</td>
<td>1550</td>
<td>~10,000 ³</td>
<td>70</td>
<td>0.7</td>
<td>[135]</td>
</tr>
<tr>
<td>SU-8/SiO₂</td>
<td>1.57/1.44</td>
<td>500</td>
<td>4, 1.8</td>
<td>1550</td>
<td>24,000 ³</td>
<td>26</td>
<td>0.62</td>
<td>[70]</td>
</tr>
<tr>
<td>SU-8/SiO₂</td>
<td>1.57/1.44</td>
<td>200</td>
<td>100, 1.5</td>
<td>1550</td>
<td>3,000 ³</td>
<td>86</td>
<td>0.26</td>
<td>[120]</td>
</tr>
<tr>
<td>SU-8/Teflon</td>
<td>1.57/1.3</td>
<td>64</td>
<td>5, 1.5</td>
<td>1550</td>
<td>24,000 ³</td>
<td>n/a</td>
<td>n/a</td>
<td>[122]</td>
</tr>
<tr>
<td>SU-8/Teflon</td>
<td>1.57/1.3</td>
<td>55</td>
<td>5, 1.5</td>
<td>1550</td>
<td>15,000 ³</td>
<td>n/a</td>
<td>n/a</td>
<td>[122]</td>
</tr>
<tr>
<td>NOA61/UFC 170</td>
<td>1.545/1.49</td>
<td>660</td>
<td>5, 1.5</td>
<td>1550</td>
<td>42,000 ³</td>
<td>n/a</td>
<td>n/a</td>
<td>[122]</td>
</tr>
</tbody>
</table>

¹Unknown if this measurement was made in air or in water.
²Measurement was made in water.
³Measurement was made in air.
⁴Measurement medium was neither air nor water.
⁵Initial Q factor measured in water for this device. The Q factor deteriorated after several experiments in the EWD system. The Q factor measured from the spectra for an oil and water mixed medium appearing in the referenced source was 17,000.
⁶Length of the straight sections in a racetrack resonator.

One of the devices designed, fabricated, and tested in this work (EBL_Test3) has the highest FOM for an SU-8 microresonator probed at a wavelength of 1550 nm. The one
example in the table of a better performing SU-8 microresonator was probed at 1310 nm [106]. At 1310 nm, the material absorption of water is about one-tenth the value at 1550 nm [110] and the material absorption of SU-8 is about one-half [136]. These factors alone would at least double the achievable FOM at 1310 nm, according to equation (61), neglecting surface scattering and coupling losses. Additionally, that microresonator was fabricated on the smooth surface of a thermally oxidized wafer, which would reduce its scattering loss compared to the microresonators in this work.
3. Design

3.1 Optical System Design

3.1.1 Transmission

3.1.1.1 Input Coupler

The input coupler includes the input waveguide facet, the input waveguide, and the adiabatic taper. The input coupler was designed to maximize coupling efficiency from a single-mode optical fiber (Corning SMF-28) to the embedded input channel waveguide, to convert the modes in the wide input waveguide to the single-mode supported by the much narrower throughput waveguide with minimal optical loss, and to minimize the width of the input waveguide for compatibility with the fabrication process utilized to form the waveguides.

3.1.1.1.1 Coupling Efficiency of Single-Mode Fiber vs. Input Waveguide Width and Height

A critical component of the optical system is the coupling efficiency between the single mode silica fiber (Corning SMF-28) and the input waveguide. The coupling efficiency at this junction sets the maximum power budget for the rest of the optical system. Proper design of the input waveguide is especially critical for the devices fabricated for this thesis, because the fabrication process employed to form the bus waveguides results in an etch depth that increases with feature width. Thus, if the input waveguide is made too wide, the etch depth of the SU-8 resist into the SiO2 channel could be so deep that the input waveguide would not support any guided modes.
Because of this concern, the coupling efficiency of the single-mode fiber was investigated with the beam propagation method (BPM) in order to determine suitable dimensions for the input waveguide. The fiber mode was simulated with a fiber mode field with the properties given for the Corning SMF-28 fiber [116]. The waveguide top claddings consisted of a 500 nm interlayer dielectric of SiO$_2$ and air above that layer. The substrate was SiO$_2$. The Fresnel reflection was neglected, because it was expected to be small relative to the overlap integral portion of the coupling efficiency. The results indicated that coupling efficiency would likely be above 10%, even for waveguides that have been deeply etched, and varies by 20% in the width and height range explored, excluding input waveguides with a width of less than 4 μm. The behavior of the coupling efficiency can be explained in terms of the effective waveguide height, a concept relevant to the analysis of slab waveguides [115, 131]. The effective waveguide height is defined as the waveguide core height plus the inverse of the exponential field decay coefficients of the fields above and below the waveguide core [115]. The overlap integral between the fiber mode field and the input waveguide mode field is expected to be positively correlated with the effective waveguide height when the effective waveguide height is less than the fiber diameter in the range of waveguide heights explored (0.4 – 1.6 μm), because the fiber mode field is about the same size as the fiber core, which is 8.2 μm in diameter. At some point near the waveguide height cut-off of the input waveguide’s fundamental mode, the effective waveguide height begins to
increase, rather than decrease, as the waveguide height is reduced. Therefore, a very thin input waveguide can potentially be effective for input coupling efficiency as well as tall input waveguides (greater than about 1.4 μm in height for this case). The concept of using a very small input waveguide with a weakly confined guided mode in order to optimize mode conversion from a fiber to a single-mode waveguide was previously explored for a Si waveguide, which achieved a 3.3 dB coupling efficiency of a standard single-mode fiber end fire coupled to a single-mode Si waveguide [137]. The same concept is applicable to the waveguides utilized in this thesis work.

Another important conclusion that can be drawn from these results is that diminishing returns in increases in coupling efficiency versus waveguide width are apparent beyond about 6 μm in width. The estimated input coupling efficiency for the range of heights and widths explored for input waveguides greater than 6 μm in width varies from about 33% to 56%, which would be more than sufficient for device testing.

To determine an appropriate width for the input waveguide, this coupling efficiency analysis was considered along with the estimated etch depth versus feature width and a target bus waveguide height of 1 μm. The selection of the target bus waveguide height is described in more detail in section 3.1.1.3.3: Phase Matching. The target width for the input waveguide was chosen to provide the best coupling efficiency with the smallest width input waveguide in order to minimize the etch depth. For an input waveguide with a 8 μm target width, the etch depth was estimated to be about
300-400 nm average from previous work. For the fabricated devices reported in this thesis, the waveguide height was estimated to be about 720 nm on average (see 4.2.9: VII. Bus Waveguide Formation / SU-8 Etch Back), corresponding to an etch depth of about 280 nm. Thus, the wide and thin input waveguide with target dimensions of 8 μm width and 1 μm height was expected to have good coupling efficiency with an SMF-28 optical fiber.

3.1.1.1.2 Mode Conversion from Input Waveguide to Throughput Waveguide

The tapered waveguide section performs the function of converting the guided mode/modes from the wider input waveguide to the modes of the bus waveguide. The mode conversion becomes asymptotically adiabatic as the tapered section is made longer [137]. The taper was designed using spline curves to avoid any abrupt changes or sharp corners in the waveguide cross section along the length of the taper. The taper section was also designed to be 2.5 mm long, which corresponds to about 1600 wavelengths at 1550 nm, to minimize propagation loss due to the varying waveguide width. A long taper length was not a problem for the system design, because long optical propagation paths (2-2.5 cm in length), formed by the drop and throughput waveguides, were needed to accommodate the microfluidics.
3.1.1.2 Bus Waveguides

3.1.1.2.1 Width and Height

The effects of varying the width and height of the bus waveguide are primarily changes in the propagation loss due to surface scattering from the rough waveguide surfaces and the change in the phase constant of the bus waveguide guided mode, which affects the coupling between the microresonator waveguide and the bus waveguide. Coupling will be covered in more detail in the following section.

The basic trends and dependence on physical parameters of surface scattering loss can be determined by considering a slab waveguide model of bus waveguides. Surface roughness in dielectric waveguides is often found to be well described by an exponential autocorrelation function [138]. For a slab waveguide with surface roughness having an exponential autocorrelation function, the surface scattering loss is described by [138, 139]

\[ \alpha_{ss} = 4.34 \frac{\sigma^2 \lambda}{2\pi \sqrt{2d^4 n_{co}}} g \cdot f, \]  

(63)

where \( \alpha_{ss} \) is the surface scattering loss in dB per unit length, \( \sigma \) is the root-mean-square (rms) surface roughness, \( \lambda \) is the free space wavelength, \( d \) is the waveguide half width, \( n_{co} \) is the refractive index of the waveguide core, \( g \) is a function of the waveguide geometry, and \( f \) is the integral of the surface roughness spectral density function. Both \( g \) and \( f \) take on values between 0 and 1. With application of the EIM, equation (63) can be applied to channel waveguides [138]. From equation (63), it is readily apparent that the
surface scattering loss is strongly dependent on \( d \), which is the half-height of a slab waveguide. If the channel waveguide is modeled as two superimposed, perpendicular slab waveguides, as is done with the EIM, then \( d \) can mean either the half-width or the half-height of the channel waveguide. Thus, increasing the channel waveguide width or height greatly reduces the surface scattering loss. The surface scattering loss is also strongly dependent on the surface roughness of the waveguide. For the devices fabricated for this thesis, the waveguides were fabricated in channels etched into a layer of \( \text{SiO}_2 \). The surface roughness of the \( \text{SiO}_2 \) channels was mitigated by utilizing a short wet etch to smooth any rough features present on the channel surfaces.

One feature of surface roughness not readily apparent from equation (63) is the dependence of the surface roughness on the refractive index contrast between the waveguide core and the cladding. A different model of surface roughness by Tien et al. yields the following equation for surface scattering loss [124]:

\[
\alpha_{ss} = \frac{4\pi^2 \sigma^2}{\lambda^2} \left( n_{co}^2 - n_{cl}^2 \right) \sqrt{\frac{n_{co}^2}{n_{eff}^2} - 1} \left( E_1^2 + E_2^2 \right),
\]

(64)

where \( E_1 \) and \( E_2 \) are the electric fields at the left and right walls of a waveguide, \( n_{cl} \) is the cladding refractive index, and \( n_{eff} \) is the effective index of the guided mode. Here, the refractive index difference is explicitly present. A convenient and positive consequence of utilizing bus waveguides made with SU-8 polymer (\( n=1.569 \)) clad with \( \text{SiO}_2 \) (\( n=1.444 \)) is that the refractive index contrast between the waveguide core and cladding is relatively small compared to an air clad SU-8 waveguide or for a waveguide core made
with a higher refractive index material, such as silicon or silicon nitride. Equation (64) provides another way to think about the surface scattering loss variation with waveguide cross-section (width and height): the smaller the waveguide is, the more field intensity will be present at the waveguide surfaces. Greater field intensity at the waveguide surfaces results in increased optical power coupled into the leaky or radiating modes by the rough surfaces of the waveguide. The intensity of the electric field at the waveguide surfaces was mitigated by utilizing waveguide dimensions which left a buffer between the effective refractive index of the fundamental mode and the cut-off refractive index (1.444).

3.1.1.3 **Microresonator-Bus Waveguide Coupling**

3.1.1.3.1 *Geometry: Vertical vs. Lateral Coupling*

In general, coupling between bus waveguides and microresonators can take place in either or both the vertical and the lateral directions. Laterally-coupled resonators typically require coupling gaps of less than 2 μm. To fabricate these structures, only a single photolithography step is required, minimizing fabrication cycle time and, concomitantly, fabrication cost. Another advantage of the laterally-coupled structure is that the microresonator is formed on the smooth surface of the substrate material, rather than the potentially more rough surface of an interlayer dielectric material, which improves sensor LOD by minimizing the surface scattering loss. A disadvantage of the laterally coupled structure is that small gaps are difficult to produce
using standard photolithography, which performs inconsistently for patterned dimensions smaller than about 2 μm (i.e. the pattern transfer fidelity becomes highly dependent on the properties of the photoresist film and the detailed photolithography [140]). Another disadvantage is that co-planar integration of the optical system limits the design flexibility for integration with other components, such as microfluidics systems. Laterally coupled systems generally leave the coupling region exposed to analytes and the bus waveguides exposed to the microfluidics gasket materials, which can generally lead to optical losses from the out-coupling of light into the gasket materials.

The vertically coupled configuration, while more complicated to fabricate, offers some general advantages over lateral coupling and some advantages in particular for integration with an electrowetting microfluidic system. The disadvantages of the vertically coupled geometry include an increased number of fabrication steps, which increases fabrication cost, and a greater potential for the development of surface roughness on the surface on which the microresonator is patterned, which worsens sensor LOD. General advantages of vertical coupling over lateral coupling include finer control of the coupling [141], due to the high accuracy of the gap dimension when controlled by material deposition, insensitivity of the coupling efficiency to small misalignments [141], increased maximum possible coupling efficiency [124], isolation of the coupling regions from analytes [87], and mitigation of interaction between the bus
waveguides and fluids in the microfluidic channels and materials used for the microfluidic gaskets. Because the gap is defined by the deposited thickness of an interlayer dielectric, which can be finely controlled in the range of 0 to 2 μm, the microresonator can potentially be fabricated completely with low cost photolithography. Furthermore, side effects of embedding the bus waveguides in an interlayer dielectric, which is needed to produce the vertical gap between the bus waveguides and the microresonator, are increased input/output coupling efficiency and reduced scattering loss in the bus waveguides [141]. A particular advantage of the vertically-coupled structure for integration with electrowetting-on-dielectric (EWD) microfluidics is, if the microresonator is to be built into the top plate, the ability to apply the conductive ground plane and the hydrophobic fluoropolymer layer with minimal considerations for the interference of these layers with the optical system, and vice-versa. The conductive ground plane material used for devices fabricated for this thesis was indium-tin oxide (ITO), which is known to be significantly absorbing around a wavelength of 1550 nm. If the interlayer dielectric were made thick enough, the ITO would not need to be patterned to minimize the optical loss of the bus waveguides. Additionally, if the surface on which these films are applied is flat, then special considerations for step coverage issues do not need to be taken. For example, if a thin ITO film less than one-tenth of the waveguide height is coated over a waveguide, the film would likely be discontinuous, resulting in an open ground plane connection. For the hydrophobic
coating, incomplete coverage of a step could result in droplets attaching to exposed hydrophilic regions on the edges of the steps. These potential problems can be completely avoided by using a vertically-coupled geometry in which the top surface of the interlayer dielectric is well planarized over the bus waveguides. For these reasons, the vertical coupling geometry was utilized for the devices fabricated for this thesis.

3.1.1.3.2 Coupling Efficiency

The interlayer dielectric thickness, or gap, has implications on the input and output coupling efficiency and the coupling efficiency between the bus waveguide and the microresonator. The input/output coupling efficiency is affected, because changes in the thickness of the interlayer dielectric also modify the refractive index profile of both the input waveguide facet and the output waveguide facet. The thickness of the interlayer dielectric defines the gap between the two waveguides, which modulates the coupling efficiency. Analytical coupled-mode theory, while inaccurate for channel waveguides, nonetheless provides a good conceptual basis to study the coupling between the bus waveguide and the microresonator waveguide. Using the coupled-mode theory for asymmetric slab waveguides from with the EIM, the trends in the coupling efficiency between two channel waveguides versus gap and versus coupler length can be mapped [115]. In the case of a circular microresonator waveguide, the coupler length is related to the radius of curvature. A larger radius leads to a longer coupler length.
Analytical coupled mode theory predicts that the coupling efficiency is periodic in length and that coupling efficiency generally decreases with increasing gap, but not necessarily monotonically. While numerical methods can be used to accurately calculate coupling efficiency ($\kappa^2$) between a straight and a curved channel waveguide, it was more expedient to fabricate devices with varying interlayer dielectric thicknesses to determine an optimum gap, rather than to theoretically predict the optimum gap.

To determine a suitable interlayer dielectric thickness, which is a thickness that balances high Q factor with an acceptable on-resonance transmission at the drop port, test sensors were fabricated on standard SiO$_2$ (thermal oxide)/Si wafers. For the test sensors (EBL_Test1-3), the best results were obtained with a microresonator waveguide width of 1.5 $\mu$m. A suitable vertical gap between the bus waveguides and the microresonator of 1.4 $\mu$m was determined empirically by varying the interlayer dielectric thickness of fabricated devices and by measuring the variation of the on-resonance transmission at the drop port with the interlayer dielectric thickness. Because the top plate embedded sensors (PL_TopPlate1 and EBL_TopPlate1) tended to have 5-15 dB greater insertion loss than the test sensors and the power measurement noise increased significantly below about -50 dBm with the measurement system utilized, a target on-resonance transmission at the drop port of greater than or equal to -35 dBm was set for the test sensors. For the EWD top plate embedded sensors, which were fabricated on a
Pyrex substrate, the intent was to replicate the best dimensions found for the test sensors.

3.1.1.3.3 *Phase Matching*

Phase matching between two waveguides is necessary to achieve optimal coupling, but there is discussion about how important it is compared to other design objectives. Some authors state that the effective index mismatch between the bus waveguide mode and the microresonator waveguide mode must be within one-thousandth of a RIU to achieve non-negligible coupling [122], whereas others state that while a large mismatch (several percent) reduces maximum coupling efficiency, coupling efficiencies greater than 10% can still be achieved [126]. In theory, the coupling efficiency is non-negligible even for waveguide modes that are significantly phase mismatched. For this thesis, phase matching was not as important a factor in determining the interlayer dielectric thickness and the microresonator waveguide dimensions, but rather, optimizing for high input/output coupling efficiency and low propagation loss are more critical, while matching the phase constants as closely as possible.

The phase constant, or effective index, of the fundamental microresonator waveguide mode that maximizes sensitivity is near cut-off, which is determined by the refractive index of the SiO₂ (1.444). The bus waveguide cut-off effective index is also defined by the SiO₂ cladding. Thus, the bus waveguide should be designed, such that its
fundamental TE or TM modes are close to cut-off to ensure good phase matching to a microresonator waveguide designed for high sensitivity. This design can be done using curves of the effective refractive index of the TE mode versus waveguide width and height for the bus waveguide and the microresonator waveguide. Figure 9 shows plots of the effective refractive index for the TE fundamental mode of the bus waveguide with a 500 nm interlayer dielectric, the bus waveguide with a 1400 nm interlayer dielectric, and the microresonator waveguide calculated using the beam propagation method (BPM) in RSoft software.
Figure 9. Effective refractive index versus waveguide width and height for the bus waveguide and the microresonator waveguide. (a) Bus waveguide (rectangular) with a 500 nm interlayer dielectric. (b) Bus waveguide (rectangular) with a 1400 nm interlayer dielectric. (c) Microresonator waveguide.

For both the 500 nm interlayer dielectric and the 1400 nm dielectric cases, the fundamental TE mode of a bus waveguide with a width and a height of 1 μm has an effective refractive index of 1.46 and 1.47, respectively. Over this range of interlayer dielectric thicknesses, the effective index of a bus waveguide with these dimensions does not change significantly. The range of interest for high sensitivity microresonator waveguide dimensions was about 1.5-2 μm in height and 1-1.5 μm in width, based on an analysis of the sensitivity (see section 3.1.2.2). These dimensions correspond to effective
refractive indices ranging from 1.451 to 1.494 for the fundamental TE mode, according to the curve of Figure 9c. In order to phase match with microresonator waveguides in this range of dimensions, target dimensions for the bus waveguide of 1 μm in width and 1 μm in height were utilized for the electron beam lithography (EBL) fabricated microresonator devices.

3.1.1.3.4 One vs. Two Waveguide Coupling and Throughput vs. Drop Port

The transmission of the microresonator is affected by the number of coupled bus waveguides as well as by the coupling coefficients. The magnitude of the coupling coefficients is related to the distance between the bus waveguide and the microresonator, the length of the coupler, and the difference between the phase constants of the guided modes in the microresonator waveguide and the bus waveguide, referred to as the phase mismatch. In this thesis, the investigation of the coupling between the bus waveguides and the microresonator is limited to the relationship between the vertical distance separating the waveguides (the gap) and the coupling coefficients.

The general tendency of the cross-coupling coefficient (κ) is to decrease as the gap is increased, because the overlap of the guided mode fields in the waveguides is reduced. As κ decreases, the loss from coupling to the bus waveguides decreases, which leads to an increase in Q factor and an improved LOD. However, the improvement in LOD obtained by decoupling the microresonator from its bus waveguides is limited by
the magnitude of the detector noise relative to the extinction ratio for the throughput port and the on-resonance transmission for the drop port. Figure 10 illustrates the trade-off between the extinction ratio and the transmission in terms of the cross-coupling coefficient ($\kappa$) for a single-waveguide coupled microresonator and a symmetric, dual-waveguide coupled microresonator, calculated using the equations from section 2.5.1. To facilitate relevance to actual measured device performance, the curves were generated using the round-trip loss coefficient ($a$) measured for EBL_Test3. Only the symmetric coupling case is considered here, because the devices fabricated and tested for this thesis are designed to have equal coupling coefficients for both bus waveguides. Several assumptions were made in the calculations. For both the single-waveguide coupled and the dual-waveguide coupled cases, the round-trip loss coefficient ($a$) was set equal to that measured for EBL_Test3 (0.7893), assuming symmetric coupling. The vertical black line marks the properties of EBL_Test3, which is a dual waveguide coupled microresonator with a gap of 1400 nm and an estimated cross coupling coefficient ($\kappa$) of 0.303 (self-coupling coefficient ($t$) of 0.953), assuming symmetric coupling. The horizontal black lines in (a) and (b) indicate the limit of detection of the resonance (LOD$_r$) for the resonance at the throughput port, based on a detector noise of 0.15 dB. The horizontal black line in (d) marks the estimated minimum on-resonance transmission (-33.2 dB) for EBL_Test3 before transmission on-resonance at the drop port drops below -50 dBm.
Figure 10. Extinction ratio and on- and off-resonance transmission loss versus cross-coupling coefficient $\kappa$. (a,c) Single waveguide coupled microresonator. (b,d) Dual waveguide coupled microresonator.
The intensity noise in the spectral measurements limits the minimum extinction ratio required to be able to detect the resonance spectral feature and it also limits the minimum on-resonance transmission through the drop port of the microresonator. For the measurement system that was utilized to test microresonators in this system, the intensity noise was estimated to be between about 0.05-0.15 dB from the high frequency noise superimposed on the resonance spectral features in measured spectra for EBL_Test2 and EBL_Test3, compared to model curves for the resonances (see Figure 48, Figure 49). This estimate agrees well with the specified typical power flatness versus wavelength of 0.15 dB for the HP 81680A tunable laser used in the measurements [142]. Therefore, for the purpose of estimating the LODr, 0.15 dB was utilized as a reasonable estimate for the intensity noise in measured spectra.

For a resonant spectral feature to be detectable, its peak point must differ from its base point by a certain amount. A reasonable estimate for the amount of this difference can be obtained by considering the limit of detection as defined by the International Union of Pure and Applied Chemistry (IUPAC) standard for the limit of detection in analysis. Using the IUPAC standard as a basis for defining the LOD of a resonance feature, the LODr is derived as follows. The LOD for a power measurement \(P_2\) relative to the baseline power measurement \(P_1\) is given by

\[
P_2 = P_1 + 3\sigma_p, P_2 > P_1
\]

(65)
where \( P_1 \), the baseline power level, is taken to be the lesser of the transmitted power off resonance or the transmitted power on resonance, which corresponds to the peak or the base of the resonance spectral feature, \( P_2 \) is the smallest measurable power measurement relative to and greater than \( P_1 \) and also corresponds to either the peak or the base of the resonance spectral feature, and \( \sigma_p \) is the noise at the \( P_1 \) power level given as a standard deviation. The relationship between the intensity noise in dB and the linear intensity noise at a given power level is

\[
\sigma_p = P \left( \frac{I_{\text{noise}}}{10^{10}} - 1 \right),
\]

where \( P \) is the measured power and \( I_{\text{noise}} \) is the intensity noise. Because a resonant spectral feature is defined by both a peak point and a base point, the LOD is defined as a minimum extinction ratio. The extinction ratio (ER) for a resonance spectral feature is given by

\[
ER = \frac{P_2}{P_1}.
\]

Using equations (65) - (67), LOD is derived as

\[
LOD_r = 3 \times 10^{\frac{I_{\text{noise}}}{10}} - 2.
\]

Equations (19), (30), and (36) define the extinction ratio for the throughput port for the single-waveguide coupled case and the drop and the throughput port for the dual waveguide coupled case, respectively. The LOD was calculated for the throughput port for both the single waveguide case and the dual waveguide coupled case and was
plotted in Figure 10a and b along with the theoretical extinction ratio curves for EBL_Test3.

If the extinction ratio is below LOD, the resonance will likely not be detectable. Therefore, the cross-coupling coefficient must be set to ensure that the resonance is at least detectable at the desired measurement port. A lower limit for on-resonance transmission from the throughput coupler to the drop coupler can be set based on the optical system power budget, transmission factors, and the estimated intensity noise. For the measurement system utilized to test devices for this thesis, the intensity noise was observed to begin to increase significantly for measured power measurements below -50 dBm. Using -50 dBm as a lower limit for the on-resonance transmitted power at the drop port and the power budget and transmission factors for EBL_Test3, the minimum on-resonance throughput coupler to drop coupler transmission was estimated to be about -33.2 dB. For EBL_Test3, which is a good representation of the other devices tested for this thesis, this minimum transmission corresponds to a minimum cross-coupling coefficient of about 0.0706 for the drop coupler. For the throughput coupler, the cross-coupling coefficient corresponding to the resonance LOD is 0.109 for the single-waveguide coupled case and 0.111 for the dual-waveguide coupled case, both of which are greater than that for the drop coupler. Therefore, according to this analysis, there is an advantage to utilizing the drop port waveguide in terms of the minimum cross-coupling coefficient and, concomitantly, the maximum Q factor, that can be
achieved by designing the device to be measured from the drop port. This analysis also shows that the minimum $\kappa$ for the drop port can be decreased further by reducing the bus waveguide propagation loss and improving the input/output coupling efficiency, thereby improving the power budget for the microresonator transmission.

For the test microresonators, the cross-coupling coefficient was not minimized to the absolute limit, because the microresonator devices fabricated on glass substrates were generally observed to have a 5 to 15 dB larger insertion loss than the test devices, which were fabricated on silicon substrates. A safety buffer in the power budget was left to ensure functionality in the case of fabrication errors or other unexpected optical losses as well as for the expected optical losses of the top plate integrated sensors. The cross-coupling coefficient $\kappa$ for EBL_Test3 is the lowest $\kappa$ for the devices tested for this thesis. For this value of $\kappa$, the intensity noise in dB observed in the spectral measurements at both the drop and the throughput port was about the same. Therefore, either port could be utilized equally well for measuring the resonant wavelengths in all of the tested devices, which had larger values of $\kappa$ than EBL_Test3.

Although the noise performance of the throughput port and the drop port for the devices measured for this thesis was ostensibly equivalent, all of the sensing measurements performed for this thesis were done using spectral measurements made from the drop port. This choice was initially driven by the observation that the on-resonance transmission for the drop port was higher than the on-resonance transmission
for the throughput port, which was true for devices that had relatively large cross-coupling coefficients. As the gap between the bus waveguides and the microresonator waveguide was increased, which decreased $\kappa$, this observation no longer applied. However, measurement from the drop port was advantageous in that the Q factor could be more easily measured from the drop port spectrum. Additionally, spectra measured at the drop port typically exhibited higher extinction ratios than spectra measured at the throughput port.

There is a prevalent notion among researchers in the field of microresonator sensors that a higher extinction ratio, preferably greater than 10 dB [105], is better for sensing.

### 3.1.2 Sensitivity

#### 3.1.2.1 Effects of Refractive Index Contrast and Waveguide Width, and Height

The refractive indices of the materials comprising the microresonator waveguide have implications on the sensitivity and the bend radius. In particular, the refractive indices of the materials comprising the waveguide define the maximum attainable sensitivity of the microresonator and the bending radius for low loss.

The maximum achievable waveguide sensitivity for a waveguide sensor (a microresonator is a type of waveguide sensor), which is defined as the ratio of the change in the mode effective index to the change in the refractive index in the waveguide cladding ($\delta n_{\text{eff}}/\delta n_{\text{cl}}$), depends on the refractive index contrast between the
waveguide core and the surrounding cladding materials [131]. This term appears in equation (55) for the microresonator sensitivity in terms of the refractive index in the sensing region, which is conventionally defined as the entire cladding region when referring to the microresonator sensitivity (the bulk sensitivity) in general. The relationship between the refractive index contrast and sensitivity can be rigorously derived for the case of a slab waveguide, because Maxwell’s equations can be solved exactly for the properties of modes in a slab waveguide. It has been shown that a higher refractive index between the core and cladding material leads to a greater maximum sensitivity for a slab waveguide [131]. For channel waveguides, the exact mode properties cannot be derived analytically from Maxwell’s equations, so such a theoretically rigorous approach to deriving this relationship may not be possible. However, inasmuch as a rectangular channel waveguide can be viewed as two slab waveguides oriented perpendicular to each other and superimposed, which is the model employed by the effective index method (EIM) for determining the properties of rectangular channel waveguides, this finding can be extended to the rectangular channel waveguide geometry. This view is supported by the fact that sensitivity is generally higher for microresonator sensors that utilize high refractive index materials (n>1.7) for the waveguide core (inorganic materials), than for materials with low refractive index (n<1.7), e.g. polymer materials (see Table 5).
For biosensing with waveguide sensors in aqueous media, the cladding refractive index is typically limited to a small range around that of water, which has a refractive index of 1.318 at $\lambda = 1550$ nm. Therefore, the cladding refractive index typically cannot be varied much from that of water. That leaves the refractive index of the waveguide substrate and core as variables. The effect of these variables on the sensitivity of a channel waveguide can be investigated using equations (57) and (58), which explicitly define the sensitivity in terms of mode effective indices, and the effective index method to solve for the effective refractive indices.

The effects of refractive index and waveguide width and height can be represented by generating curves, varying the refractive indices and the width and height using the explicit method, replacing the BPM mode solver with the EIM. Such curves were generated, considering PMMA ($n=1.49$ [143]), SU-8 ($n=1.569$), and Si$_3$N$_4$ ($n=1.98$ [144]) as core materials and SiO$_2$ ($n=1.444$), Cytop ($n=1.34$ [145]), and Teflon AF ($n=1.29$ [146]) as substrate materials. These curves are plotted in Figure 11 and Figure 12.
Figure 11. Sensitivity vs. waveguide height for several selected widths. (a) Varying core refractive index. (b) Varying substrate refractive index.
Figure 12. Sensitivity vs. waveguide width for several selected heights. (a) Varying core refractive index. (b) Varying substrate refractive index.
3.1.2.2 Sensitivity Design Curves

The simulation region included widths from 700 nm to 2 μm and heights from 500 nm to 2 μm, which encompassed the cut-offs and were expected to include or to at least indicate the trend towards the sensitivity maxima for both polarizations. The increment for each dimension was 100 nm. The arbitrary upper limit of 2 μm was set to limit the total simulation time to not much longer than 1 month (each waveguide simulation took about 40 minutes) using the available software tools (RSoft).

The beam propagation method (BPM) was utilized with RSoft software to simulate the mode effective refractive indices along with equations (57) and (58) to generate sensitivity curves for the TE and TM polarizations versus waveguide width and height. Parameters for the simulation were $\delta n_s = 1\times10^{-3}$ and $\delta \lambda_p = 0.1$ nm. The sensing region consisted of the entire cladding region. The complex refractive indices for the materials were defined according to the parameters given in Table 2. The waveguide bend was simulated by applying a conformal transformation to the refractive index profile in RSoft with a radius of 250 μm. The results of the sensitivity simulation are shown in Figure 13.
Figure 13. Sensitivity versus waveguide width and height. (Top) TE. (Bottom) TM.
The curves indicate that the highest sensitivity is obtained for tall and narrow waveguides for both polarizations. The curved edge of the curves are the cut-off for the fundamental mode of each polarization, which corresponds to a mode effective refractive index equal to the refractive index of SiO₂ (1.444).

The results of the sensitivity simulations can be explained using equation (59) by considering that the sensitivity is proportional to the overlap of the electromagnetic field intensity with the sensing region. For short and wide waveguides, the guided mode will tend to be pushed more into the substrate than in to the sensing region, because the substrate refractive index (1.444) is higher than that of water (1.318). For tall and narrow waveguides, the vertical confinement of the field is low enough that the centroid of the mode field remains well above the substrate and the field is able to extend significantly into the sensing region adjacent to the vertical sidewalls of the waveguide.

A maximum sensitivity for the TE fundamental mode of 214 nm/RIU was predicted for a width of 0.84 μm and a height of 2 μm. For the fundamental TM mode, a maximum sensitivity of 174 nm/RIU was predicted for a width of 0.73 μm and a height of 1.91 μm. These results indicate that the highest sensitivity for this waveguide can be reached with the TE polarization. The maxima for both polarizations corresponded to waveguide dimensions precisely on the cut-off line for the fundamental TE and TM modes. In practice, the waveguide dimensions that yield these sensitivities are impractical, because the bending loss and the surface scattering loss become excessively
large well before the cut-off of the mode. Therefore, the loss in the waveguide must be considered in addition to the sensitivity to determine the waveguide design that optimizes the FOM.

3.1.3 Q Factor

3.1.3.1 Effects of Refractive Index Contrast and Waveguide Bend Radius, Width, and Height

The refractive index contrast limits the minimum bend radius of a microresonator. Because a bend in a waveguide results in radiative losses, a limit on the minimum bend radius is set by the maximum acceptable bending loss for a particular application. The minimum bend radius is also determined by the waveguide width and height. Furthermore, the bend also affects the cut-off condition for the guided modes of a waveguide [147, 148], but the bending loss is typically large before the waveguide width, height, and radius are reduced to near cut-off conditions for a particular guided mode.

The behavior of the bending loss with respect to the refractive index contrast, bend radius, waveguide width, and waveguide height is difficult to accurately calculate using a single analytical method for a broad range of values for these independent variables. However, some general statements can be made regarding the trends in the bending loss with respect to these independent variables. Generally, the bending exponential loss coefficient ($\alpha_{\text{bend}}$) is reduced with increased refractive index contrast.
increases exponentially with as the bend radius is reduced [150, 151], and decreases with increasing waveguide width and height [151].

One important consequence of these variation of bending loss with refractive index contrast is that microresonators with large refractive index contrast can be made much smaller. Silicon microresonators on SiO₂ substrates, for example, have been demonstrated with bend radii down to 1.5 μm [152]. The bending loss for 2 μm wide SU-8 waveguides on a SiO₂ substrate, on the other hand, show significantly increasing bending loss for bending radii smaller than 75 μm [153]. The implication of this fact on diagnostic sensors made with microresonators is that more sensors can be packed into a device if the refractive index contrast is higher, allowing for the detection of more types of analytes.

An important trade-off to consider, based on the sensitivity curves and the trends of bending loss with waveguide width and height, is that the bending loss increases with decreasing width and height of the waveguide in contrast to the sensitivity, which, except near cut-off, increases with decreasing width and height. Because both the sensitivity and the bending loss increase with decreasing waveguide width and height, there is expected to be a particular waveguide width and height, or a series of widths and heights, that achieves an optimal balance between the bending loss and the sensitivity. These dimensions can be determined by considering the device
figure-of-merit (FOM), which is comprised of the bending loss and the sensitivity, as well as the material loss and the scattering loss.

3.1.3.2 Bending and Material Loss Design Curves

From the simulated effective refractive indices used for the calculation of the sensitivity curves in section 3.1.2.2, the combined material loss and bending loss was readily determined from the imaginary part of the effective refractive index. A conformal transformation was applied to the refractive index profile of the waveguide by the RSoft BPM software [147, 149], which was valid for radii much larger than the waveguide width. This condition was met, because the waveguide width was less than 2 μm for the range of widths explored and the bending radius was 250 μm. The imaginary part of the effective refractive index represented the combed loss of the material and the bending loss (\(\alpha_{\text{mat}} + \alpha_{\text{bend}}\)), because the imaginary part of the material refractive indices, given in Table 2, and a bending radius of 250 μm were included in the BPM simulations. Plots of the curves for both the TE and TM modes are shown in Figure 14. The simulation results show that the combined loss increases monotonically as the waveguide cross-sectional area is reduced and cut-off is approached. In addition to the mode cut-off line, a second line parallel at all points to the cut-off line is visible in the curve. This line can be considered to be the bending cut-off line, and is arbitrarily set to 30 dB/cm in the plotted curve. Beyond this line, the guided mode propagation loss increases exponentially and, thus, the Q factor and the FOM of the microresonator
become exceedingly small. The minimum values of the loss for both the TE and TM polarization over the range covered by the design curves occurred at the largest waveguide cross-section in the range, 2 μm by 2 μm. The minimum losses were 3.26 dB/cm and 2.97 dB/cm for the TE and TM modes, respectively. Noting that the loss of SU-8 is 1.65 dB/cm, the bending loss component of the combined loss is greater than about 1.3 dB/cm for the entire range explored.
Figure 14. Combined material and bending loss versus waveguide width and height. (Top) TE. (Bottom) TM.
3.1.3.3 Surface Scattering Loss Design Curves

Scattering loss is the most difficult loss factor to estimate, because it generally requires three-dimensional simulation of the radiation field from a waveguide with random surface perturbations typically on the order of nanometers to tens of nanometers [138, 154]. The volume current method (VCM) has been successfully applied to accurately model propagation loss from surface scattering [138, 139, 154]. In VCM, surface roughness is modeled as a current source on each waveguide surface. The power in the far field radiation pattern produced by the current sources for each surface is calculated and superpositioned to determine the propagation loss. A recently derived version of this method for use with channel waveguides with relatively low refractive index contrast requires only the field profile, the refractive index profile, and the rms surface roughness and the autocorrelation length for each surface [155]. Field profiles were obtained from the set of simulations that were done for the sensitivity curves in section 3.1.2.2. Tapping mode AFM measurements of the substrate and of the waveguide top surface on a test microresonator device (EBL_Test3) fabricated with EBL yielded rms roughness measurements of 6.0 and 5.5 nm, respectively. The autocorrelation length of the surface roughness was estimated to be about 200 nm by optical interferometry for both surfaces. The sidewall rms roughness and autocorrelation length were thus taken to be 5.0 nm and 200 nm, respectively, for the purpose of a comparison between TE and TM mode surface scattering loss. The TM
mode results can be considered to be accurate, because the effect of the estimated sidewall roughness on the calculation was small relative to that of the top and bottom surface roughness. The results of the scattering loss calculation using VCM [155] are plotted in Figure 15.
Figure 15. Surface scattering loss versus waveguide width and height. (Top) TE. (Bottom) TM.
The maxima for the scattering loss curves for the TE and TM were 28.6 dB/cm (width = 0.89 μm and height = 2 μm) and 29.2 dB/cm (width = 2 μm and height = 0.89 μm). The minima were 3.36 dB/cm (width = 2 μm, height = 0.57 μm) and 5.08 dB/cm (width = 0.72 μm, height = 2 μm). Surface scattering loss is worst for the TE mode for tall and narrow waveguides, whereas surface scattering loss is worst for the TM mode for short and wide waveguides. This result is consistent with experimental observations that the TE mode is much more affected by sidewall roughness than the TM mode [156]. The reason that this is the case is because the field intensity for TE modes tends to be much more concentrated at the sidewalls than the field intensity for the TM modes [156]. The opposite is true of the top and bottom waveguide surfaces. In the case of the vertically-coupled structure, the surface roughness at the top and bottom surfaces, which is almost always considered negligible in publications dealing with waveguide surface scattering loss [138, 139, 154, 156], is generally non-negligible and must be considered. In these structures, surface roughness in the substrate can be introduced by a variety of fabrication processes, most importantly by reactive ion etching (RIE). Notably, the region of the surface scattering loss curve corresponding to the lowest surface scattering loss for the TM fundamental mode corresponds to the region of the highest sensitivity in the sensitivity curve. The opposite is true of the TE mode. Therefore, in the case that the rms surface roughness is comparable on all four surfaces
of the waveguide and the surface scattering loss is comparable to the material and bending loss, the TM mode should exhibit the highest FOM.

### 3.1.4 Figure of Merit

#### 3.1.4.1 Figure of Merit Design Curves

It is useful to consider the FOM both with and without the effect of surface roughness for waveguide dimensions within the bending cut-off, because surface roughness is particular to the fabrication process and is variable. The FOM curves without surface roughness are plotted in Figure 16 and the FOM curves with surface roughness are plotted in Figure 17. The FOMs were calculated by first calculating the Q factor using equations (8) and (51), using the simulated exponential losses for the loss mechanisms to calculate the total exponential loss factor \( \alpha \), and multiplying the Q factors by the simulated sensitivity versus waveguide dimensions.
Figure 16. Intrinsic FOM versus waveguide width and height, neglecting surface scattering loss. (Top) TE. (Bottom) TM.
Figure 17. Intrinsic FOM versus waveguide width and height, including surface scattering loss. (Top) TE. (Bottom) TM.
Considering only bending and material loss, the FOM ranges from 4.12*10^6 nm/RIU (width = 2 μm, height = 2 μm) to 6.48*10^6 nm/RIU (width = 1.04 μm, height = 1.98 μm) for the TE fundamental mode and from 3.77*10^6 nm/RIU (width = 2 μm, height = 2 μm) to 6.28*10^6 nm/RIU (width = 0.93 μm, height = 2 μm) for the TM fundamental mode. Thus, within this range of waveguide dimensions, which corresponds to the edge of the resolution capability of i-line contact lithography (about 1-2 μm [140]), the sensor LOD would be expected to vary by less than a factor of 2.

Including surface scattering in the propagation loss, the FOM ranges from 1.39*10^6 nm/RIU (width = 2 μm, height = 2 μm) to 2.01*10^6 nm/RIU (width = 2 μm, height = 0.92 μm) for the TE fundamental mode and from ~0.64*10^6 nm/RIU (width = 2 μm, height = 0.85 μm) to 2.6*10^6 nm/RIU (width = 0.92 μm, height = 2 μm) for the TM fundamental mode. Thus, in the case that the sidewall roughness is comparable to the top and bottom surface roughness, the higher FOMs for the TE mode tend to wider and shorter waveguides, exactly opposite to the optimum dimensions considering the TE mode sensitivity alone. For the TM mode, on the other hand, the optimal dimensions of FOM match that of the sensitivity. Therefore, for the case of comparable roughness on all waveguide surfaces and surface scattering loss comparable to the material and bending loss, the TM mode is expected to achieve the highest FOM.

Target microresonator waveguide dimensions can be determined from these FOM curves. A good target height for the microresonator waveguide would be about 2
μm, because this height is close to the maximum intrinsic FOM for both the TE and the TM modes. A target width of about 1 μm would also seem to be a good choice. In practice, devices with microresonator waveguides of widths near 1 μm did not have detectable resonances in their spectra. Because of this problem, the devices fabricated and tested for this thesis had widths of about 1.2 μm and 1.5 μm.

3.2 Electrowetting System Design

The EWD system was designed such that no changes had to be made to the dimensions of the test optical system to accommodate the EWD microfluidics. This required the EWD system to be small enough to accommodate relatively short bus waveguides in order to maintain the insertion loss at the level observed in the test devices. The EWD system was also designed to have enough reservoirs for basic biosensing tests by including 2 analyte reservoirs, 1 waste reservoir, and 1 buffer reservoir. Examples of simple biosensing tests that this type of system would support are biotin-streptavidin binding [105] and DNA sensing [94]. For biotin-streptavidin binding, one of the analyte reservoirs would contain biotinylated-BSA, and the other would contain streptavidin. For DNA sensing, one of the two analyte reservoirs would contain a solution of non-complementary DNA as a control and the other would contain a solution of DNA complementary to the probe. In both examples, the buffer reservoir would contain the blank buffer solution in which the analytes are dissolved. The waste reservoir would be used to dispose of droplets that have already been exposed to the
sensor to clear the fluidic channel for the next droplet. The layout of the EWD system with the top plate containing the embedded optical system is shown in Figure 18. The design advice that I received from Dr. Fair and his group (Randy Evans, Bang-Ning Hsu, Yan-Yu (Ken) Lin, and Andrew Madison) was integral to the design of this system.

Figure 18. Scaled drawings of the integrated system in top view. (a) Full system. (b) Close-up of the gasket.

In EWD, droplets are actuated by applying a voltage to the electrowetting electrodes. The electric field modulates the surface tension of the droplet, which causes the droplet to move to the electrode to which the voltage was applied. By applying the voltages to a series of electrodes, droplets can be moved in arbitrary paths in the system, limited only by the layout of the electrodes.

A typical EWD device consists of a bottom plate and a top plate sandwiching a gasket that encloses the fluid reservoirs and flow channels. The bottom plate contains the electrowetting electrodes coated with a dielectric film and a hydrophobic layer, usually a fluoropolymer, such as Teflon or Cytop, as well as the gasket. The top plate
contains a conductive ground plane coated with a hydrophobic layer. Because the top plate does not contain any patterned components, it is an ideal location to embed sensors into EWD microfluidics systems.

The structure of the EWD microfluidic bottom plate consisted of four reservoirs with large electrowetting electrodes connected by a series of smaller, closely spaced electrowetting electrodes. These electrowetting electrodes were each individually connected to much larger control pads to which an array of contact pins could be contacted in order to control the voltages applied to the electrowetting electrodes. The small electrowetting electrodes had a pitch (placement interval of the electrowetting electrodes) of 605 μm and the gasket height was about one-tenth of that value (~60 μm). This ratio was chosen to minimize the droplet dispensing and splitting voltage [76] and to simplify fabrication of the gasket by a spin-coating process by minimizing the film thickness. The pitch of 605 μm was determined based on co-design considerations, which are discussed in section 3.3: Optical/EWD System Co-Design. The spacing between the electrodes was 5-10 μm and the electrodes were square in shape with a side length of 600 μm. The maximum number of electrodes, 32, limited by the control electronics, was utilized in the design.

The top plate was significantly modified from a typical EWD system to integrate the optical sensor system. The conductive ground plane and the hydrophobic coating, both required for the functionality of the EWD system [76], were retained, but with a
modification of the ground plane to accommodate the microresonator. A detailed discussion of these parts is given in section 3.3: Optical/EWD System Co-Design. Figure 19a and Figure 19b show the top and side views, respectively, of the EWD structure with the relevant dimensional parameters.

Figure 19. (a) Top view of the microresonator integrated into the EWD system (not to scale). (b) Cross-section of the EWD top plate with integrated microresonator and the bottom plate (not to scale). Typical dimensions for the devices used for testing the EBL patterned microresonators are shown.

3.3 Optical/EWD System Co-Design

To minimize insertion loss, it was critical to design the EWD microfluidics to be small, but not so small that the sensor would be larger than a single droplet. The placement interval of the electrowetting electrodes, the pitch, was set to 605 μm to ensure that the device size would both be small enough to fit on a 2” wafer and to minimize the propagation distance of light through the optical system. The insertion loss of the optical system, which is the total loss from input to output, is significantly affected by the total bus waveguide propagation loss along a path through the optical
system, which is directly proportional to the length of the bus waveguides. Insertion loss is important for both the power efficiency of the optical system as well as the LOD. The LOD begins to increase when the measured intensity approaches the noise floor of the photodetector, because the resonant wavelength estimation precision decreases with increasing intensity noise [65], which increases significantly as the noise floor is approached. Given that the bus waveguides in this device had an estimated propagation loss of 7 dB/cm, it was important to keep the path length as short as possible to optimize the power budget. The propagation length in test devices from input to output for both the drop and the throughput was about 2.2-2.5cm. This propagation distance was retained for the top plate embedded sensor to maintain comparable propagation loss. The shape and dimensions of the gasket and the spacing and size of the electrodes were adjusted to maintain this propagation distance. Because the gasket height is typically set to a constant ratio with the electrode pitch (in this case 1:10), droplet volume is roughly proportional to the cube of the electrode pitch. Therefore, reductions in the electrode pitch also decrease the volume of droplets in the EWD system, providing gains in reagent usage efficiency as well as a reduction in insertion loss.

Generally, the microresonator sensor could be embedded in either the top plate or the bottom plate. However, because the bottom plate contains a high density of electrical connections and electrodes and the top plate in a typical EWD system has only
a coating of fluoropolymer film and a transparent conductive film, it was most straightforward to integrate a sensor device into the top plate. In general, it would be expected that both the fluoropolymer and the transparent conductive film on the top plate would need to be patterned to accommodate devices integrated into the top plate. These modifications would adversely affect the ability of the microfluidic system to move droplets onto and off of the sensor. For the particular case of the polymer microresonator sensor, the resonator could be patterned on top of the fluoropolymer film. This was made possible by utilizing a reversible, oxygen plasma induced adhesion enhancement of the fluoropolymer film in order to make the microresonator adhere to the fluoropolymer surface [157, 158]. Therefore, removal of the fluoropolymer film from the sensor area was not necessary. However, if the transparent conductor used for the ground plane, indium-tin oxide (ITO), was not removed from under the microresonator waveguide, optical losses in the cavity were severe. ITO is highly absorbing around 1550 nm, because this wavelength is close to its plasma frequency [159]. To resolve this problem, a disk-shaped region of the ITO, 550 μm in diameter and centered on the 500 μm diameter sensor’s location, was etched away from the sensor area. The ITO was left intact on the rest of the surface, because the separation between the bus waveguides and the ITO film by the SiO₂ interlayer dielectric was enough to significantly mitigate the excess propagation loss due to the ITO film. Droplets in the electrowetting system tend to be only slightly greater in extent than the electrowetting electrodes with which they
are actuated. Thus, the removal of a large area of the ground plane relative to the size of the electrowetting electrodes at the sensor’s location was expected to adversely affect the actuation of droplets onto and off of the sensor, because the contact area between the droplet and the ground plane would be significantly reduced. It was difficult to predict to what extent the removal of a section of ground plane would affect droplet actuation.

The potential for significant optical coupling from the bus waveguides into the gasket material was also considered in the design. The material stack of the bottom plate includes SU-8 3000 (n~1.555) [160], Parylene C (n~1.65) [161], and Cytop (n~1.34) [145]. Because the refractive index of the parylene is higher than that of the SU-8 2002 (n~1.569) used to make the microresonator, optical loss due to coupling from the bus waveguides into the gasket was explored. Fortunately, the optimization of the interlayer dielectric thickness for the microresonator sensor performance resulted in a relatively thick interlayer dielectric, which helped reduce coupling losses. A thick interlayer dielectric increased the Q factor of the sensor and simultaneously reduced the optical coupling efficiency to gasket materials in the microfluidic system. While simulations indicated that some non-negligible optical coupling would occur between the bus waveguides and the gasket, no significant change in propagation loss was experimentally observed when the gasket was placed in contact with the top plate.
4. Fabrication

4.1 Electrowetting System

The bottom plate was fabricated using a standard EWD fabrication process [162]. The bottom plate was fabricated on a 2” Si wafer by first coating the wafer with 2 μm of SiO₂ by plasma enhanced vapor deposition (PECVD). The Cr electrodes were patterned by lift-off of e-beam evaporated Cr with a negative resist (AZ5214EIR) in n-methyl-2-pyrrolidone (NMP). SU-8 3035 was spun on at 1000 rpm, patterned by photolithography, and baked at the appropriate temperatures to produce a 50-60 μm thick gasket. The gasket was patterned to define the fluidic channels and the reservoirs. The gasket also defines the spacing between the bottom plate and the top plate and serves the function of enclosing the liquids to prevent evaporation. Low-tack dicing tape was utilized to cover the electrodes prior to deposition of the Parylene C dielectric, which functions both as a dielectric and a moisture barrier between the fluids and the electrowetting electrodes. Approximately 800 nm of Parylene C was coated onto the entire bottom plate surface using a Cookson Electronics PDS 2010 LABCOTER2 vacuum deposition system. After deposition of the Parylene C, the tape was carefully peeled off of the electrodes while using the rounded edge of a razor blade handle to hold the film down at the edge of the dicing tape. A hydrophobic coating consisting of a ~50-70 nm layer of Cytop (5:1 diluted Cytop 809A) was spin-coated at 3000 rpm, then baked at 80 °C for 1 hour and at 60 °C overnight to cure. Finally, the wafer was diced using a
Dynatex DX III scribe & break tool in non-contact mode to remove the excess substrate material.

4.2 Top Plate-Embedded Microresonator

Two processes were utilized to fabricate top plate-embedded microresonators. One process utilized photolithography (PL) and the other utilized electron-beam lithography (EBL). Where necessary, the differences in the process steps are noted in the following description of the fabrication process. The process flow is graphically depicted in Figure 20. Each step of the process flow is described in detail with relevant supporting data.
Figure 20. EWD top plate-embedded microring resonator process flow.
4.2.1 I. Etch Stop and Bus Waveguide Cladding Deposition

For the PL process, the top plate was fabricated on one quarter of a 4”, 500 μm thick fused quartz wafer. A 20 nm etch stop layer of Si₃N₄ was deposited by plasma enhanced chemical vapor deposition (PECVD), followed by a 2 μm thick layer of SiO₂ by the same method using an Advanced Vacuum Vision 310 PECVD system. For the EBL process, a 50 nm layer of PECVD Si₃N₄ (etch stop) was deposited, followed by a 1 μm thick layer of PECVD SiO₂. The purpose of the etch stop was to prevent etching of the underlying glass (SiO₂) substrate during the reactive ion etching of the waveguide channels in the PECVD SiO₂ layer as well as during the subsequent clean-up etch of the channel with buffered oxide etchant (BOE). A thicker Si₃N₄ layer was deposited for the EBL processed devices to improve the uniformity of the etched channel’s bottom surface, because deep trenches were observed to form near the sidewalls of the EBL processed devices when a 20 nm etch stop layer was used. Pyrex glass wafers were utilized for the EBL process instead of fused quartz wafers simply because the surface quality of the fused quartz wafers appeared to have deteriorated (an unacceptably high density of pits were observable on the surface) since the PL fabrication runs and the Pyrex wafers were readily available.

4.2.2 II. Cr Hard Mask Deposition

After the PECVD films were deposited, a 100 nm film of Cr was deposited on the surface by electron beam (e-beam) evaporation using a CHA Industries Solution E-Beam
evaporation system. Cr was utilized as an etch mask, because of its high selectivity to the gases used to etch the SiO\(_2\) in the formation of the waveguide trenches. This selectivity was most useful for the PL process in which 2 μm deep trenches were formed. For the EBL process, the trenches were shallower, having a depth of 1 μm. However, this depth was still large compared to the thickness of the resist used for the EBL process (300 nm), so high selectivity was also important for the EBL fabrication process. Furthermore, the Cr provided a conductive surface to sweep out charge during the writing of the patterns with the EBL tool. This feature of the Cr mask prevented any problems that might have occurred due to charging of the insulating substrate materials.

4.2.3 Il. Wafer Dicing (PL Only)

For the PL process, the wafer was diced after depositing the Cr, because the photomask utilized was not designed for whole wafer processing. The wafer was diced using either a Dynatex DX III scribe & break tool or a K&S 780 dicing saw.

4.2.4 III. Cr Hard Mask Patterning

For the PL process, AZ5214EIR image reversal photoresist was utilized as a positive resist with a dose of 45 mJ/cm\(^2\) using a Suss MicroTec MJB3 mask aligner to form the waveguide patterns. For the EBL process, the waveguide patterns were written in ZEP 520, a positive e-beam resist, with a dose of 100 μC/cm\(^2\), utilizing an Elionix ELS-7500 EX E-Beam Lithography System. 1020 Cr etchant was used to etch the Cr where the resist had been removed. For whole wafer processing, this etch was allowed to proceed
for 80s before rinsing and drying. For the PL process, each device was processed individually and the etch took about 50s – 60s. A large rectangular mark was used as an indicator for the completion of the Cr etch for the PL process.

4.2.5 IV. Bus Waveguide Channel Formation

For the PL process, reactive ion etching (RIE) was used to etch trenches into the PECVD SiO₂ using 7 sccm O₂, 64 sccm CHF₃, 20 mTorr pressure with 260W RF power for 30 minutes in a Trion Technology Phantom II reactive ion etcher. The gas chemical constituents, flow rates, pressure, and RF power settings were obtained from reference [163]. The RIE etch was followed by a 1 min clean-up etch in buffered oxide etchant (BOE). After completing the etch of the channels, 1020 Cr etchant was used to remove the Cr hard mask.

For the EBL process, the same RIE etch process was used, except the etch time 12.5 minutes, instead of 30 minutes. The RIE etch was followed by a 1 min clean-up etch in buffered oxide etchant (BOE).

The RIE etching produced very poor quality trenches with a significant amount of “grass” in the bottom of the trenches, which can be seen in Figure 21a. The grass was likely caused by micro-masking from Cr redeposited into the open areas of the Cr hard mask during etching [163].
Figure 21. SEM images showing the results of an RIE and an RIE+BOE trench etch process for a photolithographically patterned device. The Cr hard mask is still present in both. (a) RIE etched trench in PECVD SiO₂. (b) RIE+BOE etched trench.

By performing a short (60s) buffered oxide etchant (BOE) clean-up etch, the grass can be completely removed (see Figure 21b), leaving a SiO₂ channel with comparatively smooth surfaces and sidewalls that have a 70 – 80° slope. Etching of the SiO₂ layer below the PECVD SiO₂ layer in which the trench is formed was prevented by the PECVD Si₃N₄ etch stop layer. 10:1 BOE was found to etch the PECVD SiO₂ at a rate of roughly 100-200 nm/min. The etch rate of PECVD Si₃N₄ is comparatively small in 10:1 BOE, being only 2.5-11 nm/min (decreases with Si content) [164]. The optimum RIE etch time was determined empirically by correlating the etch time with evaluations of the etch quality using SEM.

Initially, this process was developed for a waveguide of approximately 4 μm in width and 2 μm in depth. The process was scaled down to produce a waveguide with a
target width of 1 μm depth of 1 μm. The as-fabricated trench was wider than the target width with an average bottom width of 1.5 μm and a sidewall slope of about 80°. Figure 22 shows the profile of a trench for an EBL patterned device. The optimal RIE etch time was found to be 12.5 minutes with the same 60 second BOE etch for these smaller channels.

![SEM image of trench produced for an EBL patterned device](image.png)

**Figure 22. SEM image of trench produced for an EBL patterned device. The Cr hard mask is still present.**

Using this process flow, the trench profile apparently does not scale linearly with the waveguide dimensions and has some flaring at the top. Despite the rough appearance of the waveguide walls and the irregular channel profile geometry, the insertion loss was generally measured to be as good as or better for devices with these much smaller bus waveguides than those with the larger, photolithographically patterned (approximately 4 μm wide) bus waveguides.
4.2.6 IVa. Wafer Dicing (EBL Only)

For the EBL process, the wafer was diced just after patterning of the entire wafer in the EBL tool and just prior to removal of the hard mask. The wafer was diced using either a Dynatex DX III scribe & break tool or a K&S 780 dicing saw.

4.2.7 V. Cr Hard Mask Removal

After completing the SiO$_2$ trench formation steps, the Cr etch mask was removed with 1020 Cr etchant. This etch typically took about 1 hour to complete, instead of the 50-80 seconds needed to etch the Cr patterns. The reduced etch rate may be due to oxidation of the Cr by the RIE plasma.

4.2.8 VI. SU-8 Channel Fill

The trenches were filled with SU-8 polymer by spin-coating a layer of SU-8 2002 at 1000 rpm, which resulted in an approximately 2.7 μm film thickness. The film was pre-baked at 65 °C for 1 minute and soft baked at 95 °C for 2 minutes. After the soft bake, the film was exposed to 365 nm UV for 20s at 10.5 mW/cm² (210 mJ/cm²). The film was then baked at 65 °C for 1 minute, 95 °C for 2 minutes, and finally hard baked at 180 °C for 30 minutes.

4.2.9 VII. Bus Waveguide Formation / SU-8 Etch Back

The SU-8 film was etched down to the trench with RIE using 90 sccm SF$_6$ and 10 sccm O$_2$, 100 mTorr, 100W RF power for the time needed to clear the surface of SU-8,
which ranged from ~160s to ~180s. Typically, an overetch, corresponding to about 50-200 nm in the bus waveguide height, was needed to completely remove the SU-8 film from the SiO₂ surface, excluding the edge bead near the edges of the substrate. The overetch was necessary to remove the SU-8 film from the surface, because the SU-8 was locally planar close the waveguide trenches, but over a long range was depressed by a small amount due to the step coverage effect (i.e. the average film thickness was greater than the film thickness near the isolated trenches).

The etch back of the SU-8 film was a key step in the process, because the thickness, and thus the optical properties, of the input waveguide and the bus waveguides depended on this step. Because the RIE tool utilized to perform the RIE etch did not have an end point detection module installed, the etch depth was controlled manually by processing each device one at a time. For each device, the etch depth of control trenches was measured immediately after the etch and the etch time was adjusted as necessary for the following device, using an estimate of the etch rate of 20 nm/s, until the optimal etch time was found for the particular batch of devices being processed. The optimal etch time typically varied between 160 and 180 seconds. Control trenches were necessary for the etch depth measurement in the cleanroom, because the etch depth of the bus waveguides could not be measured directly with the profilometer, which had a tip diameter of 12.5 μm. The target etch depth for the control trenches was set based on the etch depth that corresponded to complete removal of the
SU-8 film from the surface far from the trenches. This target depth was dependent on the width and the depth of the trench. For a 200 μm width control trench, the target etch depth was 1.8±0.1 μm for the PL patterned devices and 0.85±0.1 μm for the EBL patterned devices. A diagram of the cross-section of a control trench with the etch depth labeled is shown in Figure 23.

![Etch depth measurement diagram](image)

**Figure 23. Etch depth measurement diagram.**

Figure 24 shows bus waveguides etched for 3 different etch times fabricated with the photolithography process. The target dimensions for these waveguides were 4 μm in width and 2 μm in thickness.
Figure 24. Bus waveguides for a photolithographically patterned device etched for 3 different times. (a) 220 s. (b) 195 s. (c) 165 s.

4.2.10 VIII. Interlayer Dielectric Deposition

For the PL process, 1.2 μm of PECVD SiO₂ was deposited on top of the channel waveguides. For the EBL process, 1.4 μm of PECVD SiO₂ was deposited.

The thicknesses for the interlayer dielectric that both provided a high Q factor and maintained a low insertion loss at the drop port (on-resonance transmitted power
measured to be >-35 dBm) were determined experimentally to be about 1.2 μm for the photolithographically patterned devices and about 1.4 μm for the EBL patterned devices. Figure 25 shows SEM images of the input waveguide for EBL_Test1, which had an interlayer dielectric thickness of 900 nm, and the bus waveguide for another EBL patterned device, which had a 1600 nm interlayer dielectric. From the images it is clear that the surface morphology of the SU-8 surface was transferred to the top surface of the interlayer dielectric.
Figure 25. SEM images of the input waveguide and bus waveguide embedded in SiO$_2$ for an EBL patterned device (EBL_Test1). (a) Oblique view of the input waveguide. (b) Edge-on view of the input waveguide. (c) Edge-on view of the bus waveguide.

4.2.11 IX. Conductive Ground Plane Deposition

Indium-tin oxide (ITO) (95%/5% InO$_2$/SnO$_2$) was sputter deposited onto the PECVD SiO$_2$ at 120W with 10 mTorr Ar pressure for 30 minutes using a Kurt Lesker
PVD 75 RF sputtering system, producing a layer of ITO about 70 nm thick as measured by profilometry in a patterned ITO feature.

4.2.12 X. Conductive Ground Plane Patterning

After sputtering, the ITO was patterned using AZ5214EIR as a positive resist with a 45 mJ/cm² dose and 50s of etching in 5% HCl.

The ITO functions as the ground plane for the EWD system. ITO is a conductive material with relatively low absorption at visible wavelengths for a conductive material. The absorption of ITO can vary significantly with processing parameters. One source indicates absorption in the range of 100 - 600 dB/cm in the wavelength range of 380 nm – 750 nm [159]. Because of the high conductivity of ITO, it only needs to be applied as a very thin film, which results in a low total absorption for the visible wavelengths of light passing through the thin film. The transparency of this film enables one to observe droplets in the electrowetting system through the conductive ground plane. However, ITO absorbs strongly (≈289,000 dB/cm [165]) around a wavelength 1550 nm, because its plasma frequency corresponds to a wavelength near 1550 nm [159]. Because of this large absorption, having any ITO in contact with the microresonator severely increased the resonator loss. Therefore, the ITO had to be removed from the area where the microresonator would be patterned.

Figure 26 shows the result of etching the ITO from the resonator region and the SU-8 ring resonator subsequently patterned within that region.
Figure 26. Microscope image (20X magnification) showing a 500 μm diameter vertically-coupled microring resonator patterned in an area where a 550 μm disk of ITO was removed by etching in 5% HCl.

4.2.13 Xa. Bus Waveguide Faceting

The glass substrate was diced using a Dynatex DX III scribe & break tool to produce the waveguide facets.

Coupling of light into the input port, and from the output ports into the output fibers, was achieved by using the end-fire coupling technique. The end-fire coupling technique involves simply end face coupling the cleaved tip of an optical fiber to an input or output facet of the waveguide into which light would be coupled or is coupled from, respectively. To make the waveguide facet, the device must be cleanly cut along a line through the waveguide, so that, ideally, a smooth, flat facet of the waveguide would be exposed. For devices fabricated on Si, opening a facet in the waveguide is
straightforward if the waveguide is aligned in the [100] direction of the Si crystal. The Si can be cleaved by scratching the edge of the device and carefully bending, so as to propagate a crack along the (100) face initiated at the scratch location.

For waveguides on glass, the process of creating a facet is not as straightforward. To make a clean break in glass, a straight line must be cut into the glass at the desired location of the break. This cut can be made using a diamond-tipped scribing tool, which produces V-shaped groove with a very sharp bottom. Application of the appropriate bending initiates cracking of the glass preferentially at the groove. The crack propagates through the glass and the result is a break in the glass along the groove. A Dynatex DX III scribe & break tool was used to precisely scribe in the desired break location and to break the glass along the line. In order not to damage the waveguides, the glass was scribed on the side of the glass opposite to that of the waveguide. A microscope image of two facets cleaved by this method, one with a bad cleave and one with a good cleave, is shown in Figure 27.
Figure 27. Photomicrographs of the input waveguide facet (viewed from the top) for input waveguides from two different devices on glass substrates. (a) Bad cleave. (b) Good cleave.

The insertion loss was found to correlate with the uniformity of the top surface of the waveguide near the cleave as observed by microscopy.

Generally, the insertion loss of waveguides fabricated on glass substrates tended to have about 5-15 dB greater insertion loss than the equivalent test devices fabricated on Si wafers. This difference might have been due to additional propagation loss by the ITO film in the devices fabricated on glass, or to some physical difference in the waveguide facets, such as an angle in the facet on the glass devices.

4.2.14 XI. Fluoropolymer Coating

For the PL process, a hydrophobic layer of Cytop about 50-70 nm thick was spin-coated on top of the ITO at 3000 rpm using 5:1 diluted Cytop 809M. The Cytop film was cured by baking at 180 °C for 2 minutes. For the EBL process, low tack dicing tape was applied to the alignment marks prior to spin-coating the Cytop. A hydrophobic layer of
Cytop about 50-70 nm thick was spin-coated on top of the ITO at 3000 rpm using 5:1 diluted Cytop 809A. Prior to baking the Cytop, the low tack tape was removed. The Cytop was then baked for 2 minutes at 180 °C.

A different version of Cytop was utilized for the EBL process simply due to the lack of availability of 809M and the availability of 809A. The only difference between the two types of Cytop is in the type of functional group on the Cytop monomer that promotes surface adhesion. No substantial difference in system performance between the two versions of Cytop was observed. Teflon AF could also be used for this process at a reduced cost. However, Cytop is preferred, because it is specifically designed for adhesion to surfaces for microfabrication applications by incorporating additives into the polymer. Teflon AF was used for processing earlier devices and was observed to peel off from the substrate in the form of small flakes after a few usage cycles (sensor testing, followed by IPA rinsing and drying with N₂ gas) of the device. Teflon AF could potentially adhere to the substrate as well as Cytop with a proper application of a fluorosilane adhesion promoter, combined with an optimized coating and curing process.

4.2.15 XII. Fluoropolymer Activation

After curing, the Cytop surface was activated by a light surface ashing in O₂ at 50-70 W for 2 s prior to spin-coating SU-8 2002 on the top surface. This oxygen plasma treatment temporarily renders the fluoropolymer surface hydrophilic.
The fluoropolymer Cytop 809A (or M) was used to render the device surface hydrophobic and is a necessary component of the top plate for EWD microfluidic systems. The benefit provided by fluoropolymers, their high hydrophobicity, is also their bane in fabrication. Without surface treatment, many photoresists will simply spin off of the fluoropolymer surface when they are applied by the standard spin-coating method. SU-8 is usually applied by spin-coating and it was observed to completely spin-off of the Cytop if the Cytop was not treated prior to the spin-coating to improve adhesion. Exposure of a fluoropolymer surface with oxygen plasma is known to temporarily improve adhesion on the fluoropolymer surface [157, 158]. The hydrophobicity of the fluoropolymer surface can be restored by a high temperature bake. A very brief plasma exposure of about 2-3 seconds at a power of 70-90 W and a pressure of $7 \times 10^{-1}$ mbar was adequate to activate the fluoropolymer surface for adhesion to the SU-8 film. Following the patterning of the microresonator in this SU-8 film, the final hard bake step at $180^\circ$C for 30 minutes, which completed the cross-linking of the SU-8 polymer, also returned the Cytop film surface to its original hydrophobic state.

4.2.16 XIII. Microresonator Formation

For the PL process, SU-8 2002 was spin-coated on the top surface at 3000 rpm. The ring resonator was defined in the SU-8 2002 by baking at $65^\circ$C for 1 minute, and $95^\circ$C for 2 minutes, followed by exposure of the SU-8 with the ring pattern with a dose of 40 mJ/cm$^2$ at 365 nm. After exposure, the SU-8 was baked at $65^\circ$C for 1 minute, $95^\circ$C
for 2 minutes, and developed in SU-8 developer (PGMEA) for 30s. To ensure a complete develop, the SU-8 was developed for an additional 3-5 s after the 30s develop step. To ensure complete exposure of the SU-8, the SU-8 was flood exposed for 20s at 10.5 mW/cm² (210 mJ/cm²) and baked at 65 °C for 1 minute, 95 °C for 2 minutes, and, finally, at 180 °C for 30 minutes.

For the EBL process, SU-8 2002 was spin-coated onto the top surface at 3000 rpm. After spin-coating, the SU-8 was removed from the alignment marks using swabs soaked with acetone. Prior to the e-beam exposure, the SU-8 2002 was baked at 65 °C for 1 minute and 95 °C for 2 minutes. The ring resonator was defined in the SU-8 2002 by writing a ring pattern using EBL with a dose of 5.8 μC/cm² using the circle pattern generator (corresponds to an exposure area per dot of about 30 nm X 26 nm). Note that this dose was an overexposure, requiring the annulus defining the ring resonator to be designed with a reduced width to achieve the desired width. After exposure, the SU-8 was baked at 65 °C for 1 minute and 95 °C for 2 minutes and developed in SU-8 developer (PGMEA). To ensure complete develop, the SU-8 was developed for an additional 3-5 s after the 30 s develop. To ensure complete exposure of the SU-8, the SU-8 was flood exposed for 20s at 10.5 mW/cm² (210 mJ/cm²) and baked at 65 °C for 1 minute, 95 °C for 2 minutes, and, finally, at 180 °C for 30 minutes.

Patterning of the microresonator is a critical step. To achieve the desired combination of sensitivity and Q factor, the exposure dose must be carefully tuned to
produce the desired waveguide width. The patterning method used must also produce the smoothest possible features.

For the initial top plate embedded microresonator devices, the microresonator was patterned using photolithography. The photolithographically patterned microresonators had high Q factors, but low sensitivity, because the devices were optimized for high Q, but the microresonator waveguide dimensions were not designed to maximize sensitivity. To significantly increase the sensitivity as well as the overall device figure of merit, it was necessary to pattern features smaller than 2 μm, according to the design curves in section 3.1.2.2. Also, it was necessary to be able to adjust dimensions of the bus waveguides and the microresonator waveguides to troubleshoot the device if it did not function as predicted by theory. Therefore, electron-beam lithography (EBL) was utilized to improve the sensitivity both for its ability to pattern very small features and for the ability it provides to adjust patterns as needed.

The EBL tool utilized was an Elionix ELS-7500 EX. The CAD software for this tool has a special function, the circle pattern generator (CPG), which allows one to draw smooth arcs that will be drawn in the smoothest possible manner by the electron beam tool. A flaw in the CPG pattern drawing was discovered when a dose series was performed on one of the top plate microresonator devices. A gap was observed in the CPG patterns, located at the position where the tool started and finished drawing the ring. The gap was clearly visible after developing the SU-8, but filled in after the baking
steps. A microscope image and a measurement of the gap in a sample that had already been hard-baked are shown in Figure 28.

![Image of a microscope image and a measurement of the gap in a sample.](image)

**Figure 28.** Gap in a microring resonator pattern resulting from an error in the EBL CPG pattern drawing.

To prevent these gaps from forming in the ring, the exposure time was increased so that the area in the gap would be fully exposed. With the exposure time increased, the width of the ring pattern in the EBL CAD drawing had to be reduced to achieve the desired width. The proper exposure dose was very sensitive to the substrate conditions. The dose was found to differ significantly when rings were patterned on an SiO₂ surface as compared to that when an ~50 nm thick Cytop film covered the SiO₂ surface. The dose had to be adjusted yet again when switching from a Si substrate to a Pyrex glass substrate.

Another problem that was encountered with the EBL processing was alignment of the ring with the bus waveguides. In order to see the alignment marks in the EBL system, they had to be conductive. With the devices on SiO₂-coated Si, leaving Cr on the
alignment marks was found to work sometimes, but did not consistently prevent the
SEM image in the EBL from drifting, resulting in significant misalignments. During
alignment the SEM image would drift due to charging of the substrate in the raster
direction of the SEM, which lead to significant misalignments between the
microresonator and the bus waveguides in that direction on some of the EBL test
devices. This misalignment was significant only for the microresonator alignment with
the throughput waveguide. For the top plate embedded devices, the ITO layer
eliminated the charging problem and alignment of the microresonator with the bus
waveguides was consistently accurate.

4.3 Process Development/Test Microresonators

The process development for the EBL microresonator process was completed
using devices fabricated on 4 µm thermal oxide-coated Si wafers for faster and lower
cost processing. For the fabrication of the test devices, steps IIa, IVa, and Xa were
accomplished with simple manual scribing and breaking with a pair of tweezers and a
scribing tool, instead of with a mechanized scribe & break tool or dicing saw. This
manual scribing and breaking was performed quickly and immediately following the
prior processing steps in the cleanroom without having to coat the wafer with a
protective photoresist film for offline processing with the scribe & break tool, greatly
reducing the time for this processing step. Additionally, because crystalline silicon can
be cleaved easily and very cleanly along its (100) face, high quality waveguide facets
were obtained much more consistently than with the scribe & break process used for the devices fabricated on glass substrates. Step IX was eliminated for the test devices, because the test devices did not require a conductive ground plane. Relative to the devices fabricated on glass substrates, the test devices fabricated on Si substrates were less time consuming to fabricate and waveguide facets could be cleaved more consistently, which led to better device yields.

Using the test devices, the bus waveguide etch process was developed, problems with the EBL exposure of the microresonator were resolved, and the microresonator waveguide width was adjusted in the range of 1 to 1.5 μm until the microresonator resonances could be observed in the measured spectra. The variation of Q factor and the drop and throughput transmission spectra with interlayer dielectric thickness were also explored in order to determine an appropriate interlayer dielectric thickness. The bus waveguide dimensions resulting from a fabrication target width and height of 1 μm were, on average, a height of 0.85 μm and a base width of 1.5 μm with an 80° sidewall slope (average width of 1.65 μm). During testing, an issue with the circle pattern generator routine, wherein an anisotropic exposure pattern led to an anisotropic exposure of the SU-8 polymer comprising the resist, was resolved. The anisotropic exposure led to severe anisotropy in the refractive index, which caused the device to be non-functional. After resolving that issue, the first functional device to be tested had a microresonator waveguide width of about 1.5 μm and a height of about 1.9 μm.
5. Test and Results

5.1 Measurement Methodology

5.1.1 Measurement Instrumentation

The measurement instrumentation utilized for this work to measure spectra was an HP8164A Lightwave Measurement System with an 81680A tunable laser module, an 81623A Ge photodetector module, and an 81618A optical head interface module. The HP8164A was controlled with Labview software via GPIB. Light from the tunable laser was launched into the system via a Corning SMF 28 single-mode fiber. Light was collected from the output waveguide with a Corning 62.5/125 μm multimode, graded-index fiber. The tunable laser system supported a minimum wavelength scan step of 0.1 pm and had a noise floor of -80 dBm. Most often, one of two step sizes was used with the tunable laser, depending on the desired type of measurement. To evaluate the quality of the microresonator spectrum over a wide spectral range, a step size of 0.27 pm was used to scan over the mode-hop free range of the tunable laser from 1520 nm - 1570 nm. For high resolution measurements, such as a sensing measurement, a step size of 0.6 pm over a smaller range of wavelengths was used. Usually, the selected wavelength range was that which had the highest transmission and/or the most consistent on-resonance and off-resonance transmission, depending on the type of measurement being performed. The step size of 0.6 pm was chosen, because the tunable laser’s scan rate had
to be reduced for smaller step sizes. This step size optimized measurement resolution in both time and wavelength.

5.1.2 Data Acquisition and Analysis

Large amounts of data were generated from spectral measurements during sensing experiments, typically on the order of 100 Mb. To handle this large quantity of data, software tools for rapid data access and analysis were developed. Labview software was utilized for automated control of and data acquisition from measurement devices. Data was analyzed using a Matlab GUI tool developed specifically for visualizing and analyzing spectrum over time data. These tools enable rapid analysis and visualization of the data generated from microresonator sensing experiments.

The analysis software tracks particular points over time, referred to as monitor points. The basic algorithm for tracking monitor points corresponding to resonant wavelengths is as follows:

1. (Optional) Filter data using a zero-phase low-pass filter to eliminate high frequency Fabry-Perot or other noise.
2. Find extrema (either maxima or minima) in the data using a Van Herk running extrema filter [166].
3. Filter out local maxima (or minima) within a small range of wavelengths, usually set by the free spectral range.
4. (Optional) Curve fit with a polynomial, Lorentzian, Gaussian, or Pearson VII curves, or simply perform a zero-phase low pass filter operation, and estimate the resonant wavelengths.
5. Track peaks based on proximity to a resonant wavelength measurement in the previous frame, or based on an expected resonant wavelength shift function.

Most often, steps 1, 2, 3, 4, and 5 were performed, using a zero-phase low-pass filter prior to determining the local extrema for step 4 and frame-to-frame proximity peak tracking was utilized for step 5. The optional curve fitting in the 4th step can improve precision slightly with polynomial fitting, or can discriminate between transverse modes as well as provide parameters for calculating quality factor and finesse if Lorentzian, Gaussian, or Pearson VII curves are fitted. Exceptions to the most often utilized algorithm are noted in the following discussion of the test results when necessary.

To calculate the amount of wavelength shift produced by a change in the refractive index of the sensing region, the following phenomenological model was used to estimate the resonant wavelength shifts in the measured data:

$$\lambda_r = \left\{ \Delta \lambda_r \left[ 1 - \exp \left( - \frac{1}{t_{\text{sense}}} (t - t_0) \right) \right] + d(t - t_0) \right\} u(t - t_0) + a + bt + ct^2, \quad (69)$$

where \( \lambda_r \) is the resonant wavelength, \( \Delta \lambda_r \) is the resonant wavelength shift, \( u \) is the Heaviside step function, \( t \) is the time, \( t_0 \) is the time when analytes are initially sensed, \( t_{\text{sense}} \) is the characteristic sensing time, \( a, b, \) and \( c \) are parameters for a quadratic function, which is used to model the non-linear wavelength drift, and \( d \) is the slope of the linear drift caused by the increase in glucose concentration as the droplet evaporates. For an analysis of the sensor drift, see Appendix C: Analysis of Sensor Drift Behavior. Equation (69) is a general model that encompasses the models used to extract wavelength shifts.
from sensorgrams for experiments done for this thesis. Variations of this general model were used, depending on the experimental conditions. For experiments involving droplet merging with significant diffusion effects, which required measurement over a long time duration, the quadratic drift model was employed. For experiments involving droplet swapping with relatively small diffusion effects, which required measurement over a shorter time duration, $c$ was set to zero (i.e. a linear drift model was used). For experiments taking place in the oil medium with negligible droplet evaporation, $d$ was set to zero. Data points corresponding to transient phenomena, specifically, the dips observed in the sensorgram when swapping a droplet of glucose solution with a droplet of water, were excluded from the curve fitting.

5.2 Experiment Methodology

5.2.1 Effects of Droplet Transport on Baseline Variability

Sensing with microresonators by manipulating droplets as a means to transport analytes to a microresonator sensor has been reported [105, 120]. For sensor measurements, it is important to have a steady baseline, which is the resonant wavelength measured when no analytes are present. Variability in this baseline results in measurement error. For droplet-based transport, baseline variability can be quite large. Chao el al. state that the variability in the resonant wavelength with baseline DI H$_2$O droplet measurements was less than 50 pm [105]. Generally, baseline shifts can occur when a droplet in contact with the sensor is removed and replaced with a new
droplet (swapping), or when droplets are merged with the droplet in contact with the sensor (merging). Potentially, if all of the variables in the fluidic operations, of which droplet size and position are two examples, are carefully controlled, the variability in the baseline measurement can be minimized. Experiments were performed to evaluate the baseline variability when utilizing droplet manipulation as a means to transport analytes to the sensor. Two methods of analyte transport were evaluated: droplet merging and droplet swapping. The operations were evaluated using photolithographically patterned SU-8 polymer microresonators on SiO₂-coated Si substrates.

5.2.2 Sensing by Droplet Merging

Droplet merging in this thesis is merging a droplet with another droplet already in contact with the sensor. When the added droplet contains dissolved analyte, the analyte mixes into the blank buffer droplet by diffusion, which results in the sensor response time being limited by diffusion. An experiment was performed in which a 20 μL droplet of a 2% solution of D-glucose was merged with a 20 μL droplet of DI H₂O with both droplets brought to the sensor using a pipette. The merging operation was repeated 5 times. To refresh the solution in contact with the sensor between each trial, the mixed solution was removed with a gentle N₂ spray and a new 20 μL droplet of DI H₂O was quickly applied to the sensor using a pipette. Following the glucose merging experiments, a baseline variability measurement was performed in the same manner as
the glucose measurements by merging a 20 μL droplet of DI H₂O instead of a droplet of 2% D-glucose solution. A trace of one of the resonant wavelengths spanning all of these operations is shown in Figure 29.

**Figure 29.** Sensorgram showing 5 replicates of merging a 2% glucose droplet with a DI H₂O droplet, followed by 5 replicates of merging a DI H₂O droplet with a DI H₂O droplet. This experiment was performed with a photolithographically defined test microresonator device on a SiO₂/Si substrate. (1)- 20 μL 2% glucose droplet pipetted onto the 20 μL DI H₂O droplet. (2)- Droplet removed and replaced with a 20 μL DI H₂O droplet. (3)- 20 μL 2% DI H₂O droplet pipetted onto the 20 μL DI H₂O droplet.

In this droplet merging experiment, the baseline variability was less than 5 pm, ten times less than reported by Chao et al. [105]. The data shows an initial spike in the signal that occurs when the glucose is added, which then relaxes to the steady state baseline drift. The most likely explanation for this initial spike is that, initially, a concentrated plume of glucose solution sinks to the bottom of the droplet and comes
into contact with the sensor. This plume then diffuses throughout the droplet volume. Experiments performed using a larger volume of DI H₂O to which highly concentrated droplets of glucose solution were added showed this effect much more dramatically (i.e. much larger initial spikes were observed).

Figure 30 shows a sensorgram for an experiment that involved adding small volumes of 5% glucose-solution to a 100 μL volume of DI water to produce controlled changes in the glucose concentration.

![Graph showing sensorgram for sensitivity characterization of a photolithographically patterned test device](image)

**Figure 30.** Wavelength trace for the sensitivity characterization of a photolithographically patterned test device by merging droplets of 5% D-glucose solution with a 100 μL initial volume of water. The arrows indicate when a droplet of 5% D-glucose solution was added and the text under the arrows indicates the expected change in glucose concentration, due to the addition of the glucose solution.

Thus, to minimize these mixing transients, the use of smaller volumes of solution and of glucose solutions of lower concentration would be advisable. Completely removing the
initial water droplet and replacing it with a glucose solution (droplet swapping) reduces the initial transients from adding droplets of glucose solution. This method was investigated as well.

5.2.3 Sensing by Droplet Swapping

Swapping involves removing a droplet in contact with the sensor and replacing it with another droplet. Droplet swapping produces a faster response time than droplet merging, but is more prone to baseline variability, due to the increased number of variables involved in the swapping operation. Additional variables could potentially include fouling of the resonator surface from quickly evaporating liquid left over after a droplet is removed from the resonator and left-over materials from droplets that were removed mixing into subsequent droplets. The swapping operation was evaluated by swapping a 20 µL droplet of DI H₂O with a 20 µL droplet of 2% D-glucose solution 5 times, then swapping a 20 µL droplet of DI H₂O with a 20 µL droplet of DI H₂O 5 times. The droplet in contact with the sensor was removed with a gentle flow of N₂ gas. A trace of a resonant wavelength throughout all of these operations is shown in Figure 31.
Figure 31. Sensorgram showing 5 replicates of swapping a DI H$_2$O droplet with a 2% glucose droplet, followed by 5 replicates of swapping a DI H$_2$O droplet with a DI H$_2$O droplet. This measurement was done on a photolithographically-patterned test device on a SiO$_2$/Si substrate. 1- 20 μL 2% glucose droplet swapped with a 20 μL DI H$_2$O droplet. 2- Droplet swapped with a 20 μL DI H$_2$O droplet. 3- 20 μL DI H$_2$O droplet swapped with a 20 μL DI H$_2$O droplet.

In comparison to the merging measurement, the swapping measurement performs better. Although baseline variability was slightly worse than for the merging operation, the initial spike after application of the 2% glucose droplet was greatly reduced. Also, the estimated wavelength shifts per percent change in glucose concentration using the swapping method were consistently less than those estimated from that using the merging. This difference was probably due to the incomplete mixing of the glucose in the merging measurements. For these reasons, the droplet swapping technique was utilized to measure sensitivity of the sensor outside of the EWD system.
5.3 Sensor Measurements

5.3.1 Top Plate Embedded PL Microresonator Characterization

5.3.1.1 Sensitivity

To measure the nominal sensitivity of the photolithographically-defined top plate-embedded microresonator, the swapping measurement technique was utilized.

The sensorgram for this experiment is shown in Figure 32.

![Sensorgram](image)

**Figure 32.** Sensorgram showing 5 replicates of swapping a DI H2O droplet with a 2% glucose droplet, followed by 5 replicates of swapping a DI H2O droplet with a DI H2O droplet for PL_TopPlate1. 1- Droplet swapped with a 20 μL DI H2O droplet. 2- Droplet swapped with a 20 μL 2% glucose droplet.

Using the average of the measured wavelength shifts and the relation $1.4 \times 10^{-3}$ RIU/% for the refractive index of a glucose solution [167], the sensitivity was calculated to be about 26 nm/RIU. This sensitivity was relatively low compared to other reported
microresonator sensors, because the microresonator waveguide was not designed to maximize sensitivity. Rather, the waveguide dimensions of 4 μm width by 2 μm height were based on those previously reported in [118] for an SU-8 channel waveguide.

5.3.1.2 Quality factor and Figure of Merit

The Q factor of this sensor was determined from a spectral measurement while immersed in water. The spectrum is shown in Figure 33.

![Spectrum](image)

Figure 33. Spectrum measured at the drop port for the PL_TopPlate1 device in water.

The measured Q factor was about 24,000. Considering the sensitivity of 26 nm/RIU, the FOM for this sensor was calculated to be 0.62*10^6 nm/RIU. This FOM was improved by designing the microresonator waveguide to increase the sensitivity, using electron beam lithography (EBL) to pattern the waveguides.
5.3.2 EBL Microresonator Characterization and Optimization

5.3.2.1 Sensitivity

To improve the microresonator sensitivity, the microresonator waveguide dimensions were reduced significantly and electron beam lithography (EBL) was utilized to fabricate the smaller waveguides. Test microresonator sensors fabricated on SiO₂/Si substrates were utilized for process development and to determine an optimal interlayer dielectric thickness. For the EBL test sensor data presented herein, all devices had a microresonator waveguide width of 1.5 μm and a height of about 1.9 μm. The sensitivity for two of the test devices was characterized. The sensorgram for a glucose sensitivity measurement of EBL_Test3, which had a 1,400 nm interlayer dielectric, is shown in Figure 34.
Figure 34. Sensorgram showing 5 replicates of swapping a DI H₂O droplet with a 2% glucose droplet, followed by 4 replicates of swapping a DI H₂O droplet with a DI H₂O droplet for EBL_Test3. 1- Droplet swapped with a 20 μL DI H₂O droplet. 2- Droplet swapped with a 20 μL 2% glucose droplet.

The sensitivity for EBL_Test3 was calculated from the average of the estimated resonant wavelength shifts produced by the 2% D-glucose to be 82 nm/RIU. The sensitivity of EBL_Test1, which had an interlayer dielectric of 900 nm, was also characterized in the same manner and found to be 84 nm/RIU.

5.3.2.2 Quality Factor, Insertion Loss, and Figure of Merit

To explore the variation of Q factor and insertion loss with the interlayer dielectric thickness, three devices with varying interlayer dielectric thickness were characterized. The tested microresonators had interlayer dielectric thicknesses of 900 nm (EBL_Test1), 1,200 nm (EBL_Test2), and 1,400 nm (EBL_Test3) and all had
microresonators with waveguide dimensions of about 1.5 μm width and 1.9 μm height.

Spectra measured for these devices are shown in Figure 35.
Figure 35. Spectra measured at the drop port and the throughput port for three microresonator devices with microresonator waveguide widths and heights of 1.5 μm and 1.9 μm, respectively, and different interlayer dielectric thicknesses. (a) 900 nm. (b) 1,200 nm. (c) 1,400 nm.
These spectral measurements clearly demonstrate the effect of the interlayer dielectric thickness on the Q factor, the extinction ratio, and the insertion loss. The variations in the spectral measurements with the interlayer dielectric thickness match the predicted behavior with respect to the cross-coupling coefficient ($\kappa$), or, equivalently, the coupling loss ($\alpha_{\text{coup}}$), shown in Figure 10. For both the throughput port and the drop port, the observed Q factor increases with a decrease in the cross-coupling coefficient. For the throughput port, a decrease of the cross-coupling coefficient leads to a decrease in the extinction ratio and a decrease in the on-resonance insertion loss. For the drop port, a decrease of the cross-coupling coefficient leads to an increase in the extinction ratio and an increase in the on-resonance insertion loss.

Using the sensitivities and Q factors for each of the test devices, the FOMs ($Q^*S$) were estimated to be $0.24 \times 10^6$ and $1.2 \times 10^6$ for EBL_Test1 and EBL_Test3, respectively. $1.2 \times 10^6$ is the highest FOM reported to date for vertically-coupled microresonator devices using SU-8 polymers measured near the 1550 nm wavelength. From this testing, it was determined that a microresonator waveguide with dimensions of about 1.5 $\mu$m width as measured by SEM (FEI XL30 SEM-FEG) and about 1.9 $\mu$m height as measured by a 3D optical profilometer (Zygo NewView 5000) and interlayer dielectric thickness of 1,400 nm would be excellent for the top-plate embedded waveguide-coupled microresonator optical system. Thus, the design parameters for this device (EBL_Test3) were chosen as target dimensions for the top plate embedded microresonator device.
5.3.3 EBL Top-Plate Embedded Microresonator Characterization

5.3.3.1 Sensitivity

A top plate embedded microresonator was fabricated with the same interlayer dielectric thickness as EBL_Test3 (1,400 nm), but with a microresonator waveguide with a width of about 1.2 μm and a height of about 2.0 μm, as measured by 3D optical profilometry. The width was smaller than intended, because the EBL exposure conditions (dose and target width) expected to yield a 1.5 μm width actually yielded a waveguide of a smaller width. However, the device was functional and was interesting to characterize to compare the measured sensitivity to the theoretically predicted sensitivity.

Sensitivity was measured by performing the swapping method with 2% glucose described earlier. The sensorgram is shown in Figure 36.
Figure 36. Sensorgram showing 5 replicates of swapping a DI H₂O droplet with a 2% glucose droplet, followed by 5 replicates of swapping a DI H₂O droplet with a DI H₂O droplet. The device under test was EBL_TopPlate1. 1- Droplet swapped with a 20 μL DI H₂O droplet. 2- Droplet swapped with a 20 μL 2% glucose droplet.

Using the average of the measured wavelength shifts and the relation $1.4 \times 10^{-3}$ RIU/% for the refractive index of a glucose solution [167], the sensitivity was calculated to be about 89 nm/RIU. The sensitivity was expected to be, and is, higher in this device than in the test devices (EBL_Test1 and EBL_Test3), because of the large reduction in the waveguide width. More field extends into the sensing region in a smaller waveguide, which is expected to increase sensitivity, according to equation (59).
5.3.3.2 Quality Factor and Figure of Merit

To calculate the Q factor for this device, the Q factor was measured in water. The measured spectrum for this device is shown in Figure 37.

![Measured Spectra (gap=1400 nm)](image)

**Figure 37. Spectra measured at the drop port and the throughput port for the EBL_TopPlate1 device.**

This spectrum was similar to that of the 1,400 nm EBL process development device. The measured Q factor of 8,400 was only about half of the target value, however. One of the main causes of the discrepancy was the smaller microresonator waveguide width relative to the EBL_Test3 waveguide width, which would significantly affect the phase constant of the guided mode in the microresonator waveguide. It is likely that better phase matching between the microresonator waveguide and the bus waveguides led to an increased cross-coupling coefficient ($\kappa$) (lower self-coupling coefficient ($\tilde{t}$)), or, equivalently, an increased $\alpha_{\text{coup}}$, which would result in a lower Q factor. The self-
coupling coefficient (t) estimate was smaller for EBL_TopPlate1 (0.889) than for EBL_Test3 (0.953), which supports the hypothesis that coupling between the bus waveguides and the microresonator was stronger for the top plate embedded sensor (κ and α_{coup} were bigger). Furthermore, the internal loss estimate for EBL_TopPlate1 (20.0 dB/cm) was larger than that of EBL_Test1 (13.1 dB/cm), which means that the sum of the material, bending, and scattering losses (α_{mat}, α_{bend}, α_{sc}, respectively) was larger for EBL_TopPlate1 than for EBL_Test3. Bending loss is expected to increase slightly if the waveguide width is reduced by 300 nm and the height is increased only by 100 nm. Also, a waveguide with reduced width would be expected to have greater surface scattering losses, due to the increased overlap of the guided mode field with the sidewalls of the waveguide. Furthermore, the surface roughness might have been higher for this device than for the EBL_Test3 microresonator device, which would further increase surface scattering losses.

Considering the Q factor of 8,400 and the sensitivity of 89 nm/RIU, the FOM was calculated to be 0.75*10^6 nm/RIU. This FOM is only a slight improvement over the FOM for the PL top plate embedded sensor (0.62*10^6 nm/RIU), because of the reduced Q factor. However, given that test devices achieved a FOM of 1.2*10^6 nm/RIU, it is expected that the top plate embedded sensors would achieve significantly higher FOMs than the PL sensors with some refinements in the fabrication process to reduce the
surface scattering loss and with optimization of the microresonator waveguide dimensions.

### 5.3.3.3 Polarization Response

During analysis of the EBL test data, the importance of measuring the optical polarization utilized for sensor characterization for the purpose of matching experimental measurements to theoretical predictions became apparent. To clearly compare theory with experiment, the active polarization of the EBL defined top plate embedded microresonator sensor that was analyzed for this thesis (EBL_TopPlate1) was determined experimentally. For this device, a significant dependence of the insertion loss on the polarization was observed. To determine the input polarization (i.e. identify it as TE or TM) corresponding to the minimum insertion loss, which was utilized in the experiments, a half-wave plate was used first to rotate the input polarization, such that transmission through the device was maximized. The device under test was then removed from the system and the photodetector was placed directly in front of the input fiber. A polarizer was placed between the input fiber and the photodetector and rotated until transmission was minimized. The polarizer angle corresponding to a null in the measured power was measured to be $\sim 70 \pm 10^\circ$. The photodetector was then placed in front of the fiber connector and the polarizer was placed between the photodetector and the fiber connector. The polarizer was rotated until transmission was minimized and the angle of the polarizer corresponding to the null in the measured power was
measured to be \(-75 \pm 10^\circ\). According to the tunable laser specifications [142], the tunable laser has a polarized output with a 16 dB polarization extinction ratio and the electric field vector points in the direction of the key in the fiber connector, which corresponds to a vector pointing towards the ceiling in the lab frame of reference. Therefore, the electric field exiting the input fiber had about the same orientation as the field exiting the fiber connector. In the waveguide frame of reference, the electric field vector pointing to the ceiling in the lab frame of reference corresponds to the electric field pointing in a direction perpendicular to the substrate. This orientation matches that of the TM modes in the waveguide (see Figure 5). Therefore, the incident field in the measured orientation was expected to couple primarily to the TM modes of the optical system. The orthogonal orientation of the half-wave plate, which would orient the input polarization to couple efficiently into the TE modes of the optical system, produced no observable resonances in the drop port spectrum of EBL_TopPlate1 and overall transmission was significantly lower. From these measurements, it was inferred that the EBL defined top plate embedded devices supported only the TM mode when measured at the drop port. It is likely that the high insertion loss for the TE polarization was due to the sidewall roughness in the bus waveguides and/or the high loss of the TE mode in the microresonator waveguide.
5.4 EWD System Microresonator Testing

5.4.1 EWD-Integrated Microresonator Measurement System

The EWD with integrated microresonator measurement system consisted of the optical test equipment described previously for measurement of the optical system as well as several additional components to control the EWD microfluidic system and to monitor the droplet motion. With the generous assistance of Randall Evans and Bang-Ning Hsu of Dr. Fair’s group, I was able to utilize their electrowetting test system for experiments utilizing electrowetting-on-dielectric (EWD) microfluidics. The electrowetting test setup consisted of a microscope with a Basler CCD camera for observing droplet motion, an AC voltage source connected to a 10X voltage amplifier to supply the driving voltage, a voltage switch array interfaced with a computer via USB to control the electrodes, and two long distance translation stages to keep the droplets in view as they moved through the fluidic channels. Figure 38 shows photographs of the measurement system with the components labeled.
Figure 38. EWD-integrated microresonator measurement system. (Top) Whole measurement system view (excluding the tunable laser system). (Bottom) Close-up view of the EWD-integrated microresonator measurement system.
5.4.2 Measurement Preparation

A careful process was required to load the reservoirs of the microfluidic system, firmly clamp the top plate down on the bottom plate, and align the fibers to the top plate’s waveguide facets. First, the bottom plate was taped to a metal plate on a translation stage. Next, the top plate was carefully brought into contact with and aligned to the bottom plate by observing alignment marks through an optical microscope with a 2X objective. After aligning the top plate to the bottom plate, a Thorlabs v-mount clamping arm with double-sided tape applied to the end was lowered onto the top plate and firmly pressed, so that the tape would adhere to the top plate. The clamping arm was lifted up along its post with the top plate attached and rotated to the side, so that it no longer contacted the bottom plate.

With the top plate to the side, the gasket was covered with just enough silicone using a pipette oil to cover the area of the gasket inside of the narrow trench running around the border of the gasket. Trenches running around the edge of the gasket, which can be seen in Figure 18b, helped to contain the oil in the gasket during filling of the reservoirs and allowed the height of the oil to be raised high enough while constrained at the edges of by the reservoir, such that droplets of water could be dispensed under the oil by pipette. One of the reservoirs was loaded with 300 nL of DI H$_2$O and a second one with 300 nL of 2% D-glucose solution. Normally, the reservoirs would be loaded by pipetting solution through vias in the top plate. While it was technically possible to
define holes in quartz by using techniques such as powder micro-abrasion, deep reactive ion etching, laser etching, or mechanical drilling, the additional complications of those fabrication steps were avoided for expediency. Incorporation of hole-drilling steps into the fabrication process would have required additional process development. Because access holes were not necessary to demonstrate a prototype functional system or to characterize the integrated sensor, these steps were excluded for the initial prototypes. Pipette holes eliminate the need to perform the difficult and time-consuming process of loading the EWD system’s reservoirs. Future devices should incorporate pipette holes to facilitate more rapid device testing.

After the reservoirs were loaded, the top plate was brought into contact with the bottom plate using the clamp arm. Fine alignment was performed by gently nudging the top plate until the etched ground plane region in the top plate within which the microresonator was located was aligned to the electrowetting electrode on the bottom plate. After alignment, the end of the clamp arm was pressed firmly down firmly and the clamp arm’s locking screw was screwed tightly. The metal plate supporting the sealed bottom and top plates was unscrewed from the first translation stage and secured with screws to another 3 axis translation stage mounted to the EWD translation stage on which the input and output fibers were positioned ready for alignment. The EWD translation stage consisted of two long-distance, single axis translation stages fixed together perpendicularly, a Thorlabs lab jack was mounted on top of the long-distance
translation stages, and three three-axis translation stages mounted on top of the lab jack, one to support and align the input fiber, one to support the device under test, and one to support the output fiber.

After moving and securing the loaded and sealed microfluidic system to the EWD translation stage, the array of contact pins for the electrowetting system was aligned with the contact pad array on the bottom plate and secured with a v-mount clamp arm. Next, a wire was attached to the top plate with an alligator clip for connection to the electrical ground. After electrical connections were made, a single-mode fiber was aligned to the input waveguide facet on the top plate and a multimode fiber was aligned to the output waveguide facet. The alignment of both fibers to their respective waveguide facets was adjusted to maximize transmitted power. The power meter of the HP8164A Lightwave Measurement System (tunable laser system) was used to monitor the transmitted power during this adjustment.

After optical alignment, the EWD translation stage was moved to the electrowetting system test bench and aligned with the observation camera for a top view of the system. The ribbon cable connecting to the contact pin array was plugged into the voltage switch array, a component that enables voltage to be applied to selected electrowetting electrodes via a computer interface, and the optical fibers were connected to the tunable laser source and the photodetector. Finally, a half-wave plate, which was mounted to a Thorlabs fiber U-bench that was inserted into the beam path between the
laser output and the input waveguide facet, was used to adjust the polarization of the input light to achieve optimal transmitted power in the optical system.

### 5.4.3 EWD System Test Results

#### 5.4.3.1 Initial Testing with Photolithographically Patterned Top-Plate-Embedded Microresonators

First, a test was performed to determine the baseline variability for merging operations in the electrowetting system by merging several droplets with the initial droplet in contact with the sensor. These droplets were actuated to the sensor by applying 40 - 80V peak-to-peak AC voltage at 1 kHz frequency to actuate the droplets. Figure 39 shows a sensorgram from the measurement.
Figure 39. Sensorgram showing droplet merging in the electrowetting system with the PL_TopPlate1 device. Droplet merging times are indicated by the arrows.

The sensorgram shows negligible change in the resonant wavelength after each droplet was merged with the droplet in contact with the sensor.

Following this successful baseline test, a glucose measurement was performed by merging a 2% D-glucose droplet with a DI H₂O droplet in contact with the sensor. The glucose measurement was performed twice, once using 20 cSt silicone oil and once using 2 cSt silicone oil as the immiscible medium. The merging of a 2% D-glucose droplet with a DI H₂O droplet is depicted in Figure 40.
The amount of wavelength shift produced by the change in glucose concentration was determined by fitting a model curve to the measured data given by equation (69) with $d$ set equal to zero. $d$ was set to zero, because droplet evaporation, which was mitigated by the oil medium, was negligible over the measurement time scale. The model was fit to the data using non-linear least squares regression with Matlab’s Curve Fitting Tool, varying the parameters $t_0$, $t_{\text{rise}}$, $\Delta \lambda$, $a$, $b$, and $c$. Sensorgrams for these merging operations and model fits to the traces are shown in Figure 41.
Figure 41. Sensorgrams (black dots) for glucose measurements in the electrowetting system and model fits (red lines) to the traces. This measurement was done with PL_TopPlate1. (a) 2% D-glucose droplet merging with a DI H₂O droplet in 20 cSt silicone oil. (b) 2% D-glucose droplet merging with a DI H₂O droplet in 2 cSt silicone oil. The arrow indicates when droplets were merged.

For the 20 cSt silicone oil measurement, a resonant wavelength shift of about 21.4 pm/% was measured with a time constant ($t_{\text{sense}}$) of 2 min 49 s. For the 2 cSt silicone oil measurement, a resonant wavelength shift of about 31.5 pm/% was measured with a
time constant of 3 min 40 s. Using the wavelength shift estimated from the model fits and the reasonable assumption that the two merged droplets had equal volumes, based on the size of the droplets observed through the microscope, the estimated device sensitivity with 20 cSt silicone oil and 2 cSt silicone oil was about 15 nm/RIU and 23 nm/RIU, respectively.

The difference in sensitivity can be explained by considering the effect of the oil viscosity on the thickness of a film of oil between the droplet and the sensor. A thin film of silicone oil is present between the droplet and surfaces of the channel when silicone oil is used as an immiscible medium [76, 98]. The parameter of interest, the viscosity, is a measure of a fluid’s resistance to deformation by tensile and shear forces. When a droplet is moved beneath the sensor by electrowetting, shear and tensile forces displace and deform the oil. The result of these forces at equilibrium is the formation of a film of oil between the water droplet and the sensor surface, which has a thickness that is correlated with the oil’s viscosity.

Fortuitously, lower viscosity silicone oil is not only better for both the sensor performance, but also the EWD system performance. The threshold voltage is reduced for oil relative to that of air and is also reduced for lower silicone oil viscosities [76]. However, the vapor pressure of oils generally increases as viscosity decreases. At some point, an oil’s vapor pressure, or volatility, becomes too high for practical use. Silicone oil with a viscosity of 2 cSt was the lowest viscosity of silicone oil that could be
practically utilized in the experiments performed for this thesis. Lower viscosity silicone oils were tested, but rapidly evaporated during testing, rendering the EWD system non-functional before tests could be completed.

Glucose measurement by droplet swapping was also evaluated in the EWD system. Baseline variability was evaluated by moving a droplet of DI H₂O in contact with the sensor to an adjacent electrode, then moving it back onto the sensor. As apparent from the wavelength trace of the experiment shown in Figure 42, the baseline shift was negligible (i.e. significantly smaller than the standard deviation of the resonant wavelength estimate). On the second try, the droplet failed to move away from the sensor, but was shifted slightly from its original position. Some baseline shift was observed that might have been due to the erratic motion of the droplet that was observed in this attempt to move the droplet as well as to the slight shift in droplet’s position. Both of these effects could affect the thickness of the silicone oil film entrained between the droplet and the sensor surface, which would result in a shift in the resonant wavelength.
Figure 42. Sensorgram showing swapping of DI H₂O with DI H₂O and with 2% D-glucose and vice-versa in the electrowetting system. Measurement was on a photolighographically-patterned device (PL_TopPlate1). 1- DI droplet moved off and then back on. 2- Failed attempt to move DI droplet. 3- DI droplet moved off and glucose droplet moved on. 4- Glucose droplet moved off and DI droplet moved on.

Following the baseline variability measurements, a swap with a 2% D-glucose droplet, followed by a swap with a DI H₂O droplet was demonstrated. The swapping of the DI H₂O droplet with the 2% D-glucose droplet is shown in Figure 43.
Figure 43. Image sequence showing the droplet swap operation in order from left-to-right for the PL_TopPlate1 device integrated with the EWD system.

During testing, it was found that only a limited number of swaps were possible before the droplet in contact with the microresonator would cease to actuate properly to an adjacent electrode. For this experiment, only four droplet swaps were possible before failure to actuate was observed. Elsewhere on the electrowetting system, where the top plate had not been modified by removal of the ground plane or the addition of an SU-8 polymer ring, it was possible to actuate the droplet between two adjacent electrodes over one hundred cycles with no apparent changes in actuation performance at rates as high as 20 Hz, limited by the control electronics. Therefore, either the removal of a large area of the ground plane relative to the contact area of the droplet, or the addition of the SU-8 microresonator to the top plate, or a combination of these two factors, affected the ability actuate the droplet at the sensor electrode, the electrowetting electrode directly below the microresonator sensor. Also, the voltage required to actuate the droplet onto
and off of the sensor electrode increased with the number of cycles. Because the phenomenon appears to be cycle dependent, and because of the observed cycle dependence of the threshold actuation voltage, the build-up of charge in the Parylene C/Cytop dielectric is a likely cause of this problem. Charging of the dielectric is a well-known phenomenon, but the charging mechanism is not yet fully understood [76, 98]. The use of an AC voltage mitigates this charging, which is why a 1 kHz AC voltage was utilized to actuate droplets for the experiments that were done for this thesis. The build-up of charge limits the contact angle change that can be achieved by application of a voltage to the electrowetting electrode and, given enough charge build-up, can prevent electrowetting entirely. It is possible that the removal of the ground plane around the sensor exacerbated the charging of the dielectric, which led to an inability to wet the droplet to the Cytop surface by application of a voltage. More experiments are necessary to determine if charging really is exacerbated by the higher resistance path between the droplet and ground, due to the removal of the ground plane around the sensor, and to rule out other factors, including the presence of the SU-8 polymer ring that forms the microresonator sensor.

Model fits to the measured sensorgrams were done to estimate the wavelength shifts in the droplet swapping experiments. Close-ups of the sensorgrams and model fits for both swaps are shown in Figure 44.
Figure 44. Sensorgrams (black dots) for the glucose swapping measurements in the electrowetting system and model fits (red lines) to the sensorgrams for the PL_TopPlate1 device. (a) Swapping a DI H$_2$O droplet with a 2% D-glucose droplet in 2 cSt silicone oil. (b) Swapping a 2% D-glucose droplet with a DI H$_2$O droplet. The arrow indicates when droplets were swapped.

The wavelength shift produced by swapping droplets, taking the average of the first and second swap, was about 27.9 pm/%. This glucose sensitivity corresponds to a refractive
index sensitivity of about 20 nm/RIU. Also, the first swap had a time constant of 15 s, more than 10 times smaller than the time constant measured for the merging operation. The short sensing time is primarily due to the dramatic reduction in diffusion time necessary for the glucose to come into close proximity to the sensor surface. Close proximity can be defined as the distance from the waveguide core boundary at which the field decays to $e^{-1}$ (36.8%) of its value at the boundary. This distance generally varies with the waveguide dimensions, but was found to vary only within a small range (300 to 400 nm) for all of the waveguides tested for this thesis, according to numerically simulated field profiles and according to decay constants calculated with the slab waveguide approximation and with the EIM. In fact, the diffusion time would be expected to be zero in the case of droplet swapping, because the whole volume of water is replaced with water containing the analyte already fully dissolved and in a steady diffusion state. The observation of a small diffusion effect in droplet swapping indicates that some other diffusion effect, besides the diffusion of glucose within the water droplet due to mixing, is occurring. Possible explanations include a thin layer of water remaining present on the sensor surface when the first droplet is moved away into which the glucose from the second droplet diffuses, or that some glucose diffuses into the oil and/or the SU-8 polymer. This effect is observed both outside of the EWD system (without silicone oil) and inside it.
The reduction of sensitivity relative to the nominal sensitivity due to the thin film of silicone oil between the sensor surface and the droplet is important for comparing the effect of the immiscible medium’s viscosity on the sensor performance. Using the measured sensitivities in 20 cSt oil and 2 cSt oil and the measured nominal sensitivity, the sensitivity measured without any oil (26 nm/RIU), the 20 cSt oil reduced sensitivity to about 58% of nominal and the 2 cSt oil reduced sensitivity to about 77-88% of nominal. Considering the initial Q factor measured for the device used for the preceding glucose tests (PL_TopPlate1), which was measured to be 24,000 in water, the FOM for the photolithographically-patterned top plate embedded device was estimated to be $0.62 \times 10^6$ nm/RIU.

5.4.3.2 Testing Results with EBL Patterned Top-Plate-Embedded Microresonators

The photolithographically defined microresonator devices were successful at demonstrating the functionality of the top plate-embedded microresonator sensors in an EWD system. The sensitivity of these devices was not optimized, however, because the microresonator waveguide was not optimized for maximum sensitivity. Theoretical estimates of the microresonator sensitivity indicated that a microresonator waveguide with a narrower width would show greatly improved sensitivity. The dimensions at which significant gains in sensitivity for the fundamental TE and TM modes of the microresonator waveguide were found to be below 2 μm in section 3.1.2.2. Features with lateral dimensions smaller than 2 μm are difficult to pattern consistently using
standard i-line (365 nm) contact photolithography. So, electron-beam lithography (EBL) was employed to fabricate smaller dimension microresonators and bus waveguides. EBL provided both the ability to pattern very small features well below 2 μm and to adjust the dimensions of the features quickly, without fabricating a mask.

Top plate embedded microresonators were successfully fabricated with the EBL process and tested with the EWD system. The response to glucose was measured in the EWD system with the merging technique using 2 cSt silicone oil. An image sequence depicting the merging operation is shown in Figure 45 and the sensorgram and the model fit to the sensorgram are shown in Figure 46.

Figure 45. Image sequence showing the droplet merge operation in order from left-to-right for the EBL_TopPlate1 device integrated with the EWD system [134]. © 2012 IEEE
Figure 46. Sensorgram (black dots) for the merging glucose measurements in the electrowetting system for the EBL_TopPlate1 device and model fit (red lines) to the sensorgram. The sensorgram shows the response of the resonant wavelength before and after a 2% D-glucose droplet was merged with a DI H2O droplet. The arrow indicates when a glucose droplet was merged with the DI H2O droplet [134].

The model fit estimate of the wavelength shift was 101 pm/%, which corresponds to a sensitivity of about 72 nm/RIU. The estimate of the time constant was about 1 min 10 s. This sensitivity was three-times that of the photolithographically-patterned sensor (PL_TopPlate1) measured by droplet merging in 2 cSt silicone oil. The sensitivity of this device measured without silicone oil was 89 nm/RIU. Therefore, the sensitivity in 2 cSt silicone oil was about 80% of nominal, which agreed well with the sensitivity reduction for 2 cSt silicone oil measured with the photolithographically patterned top plate embedded sensor (PL_TopPlate1).
While the sensitivity was greatly improved for the EBL patterned device by reducing the width of the microresonator waveguide, the Q factor for the EBL patterned device was lower: only 8,400. Thus, the FOM, $0.75 \times 10^{-6}$ nm/RIU, was only marginally better than that of the high Q, low sensitivity photolithographically patterned device. According to the test EBL devices, the potential FOM for the devices fabricated with the EBL process was at least $1.2 \times 10^{-6}$ nm/RIU. The potential causes for this discrepancy are increased coupling between the microresonator waveguide (increased coupling loss) and increased internal losses, due to increased bending loss and to increased surface scattering loss. A more detailed discussion is given in sections 5.3.3 and 6.3.
6. Theory vs. Experiments

6.1 PL Defined Top Plate Embedded Microresonators

The photolithographically-defined top plate-embedded microresonator tested (PL_TopPlate1) had a width of 4 μm and a height of about 1.8 μm. The purpose of analyzing this device is to show the correspondence of the iterative method when applied to a waveguide for which the conditions for accuracy of the effective index method (EIM) are met.

6.1.1 Sensitivity

Because the width is significantly greater than the height for this waveguide, the weakly guiding approximation in the lateral direction is valid and the effective index method can be used to accurately estimate the effective index of the fundamental mode of this waveguide. Solving the resonance equation given by equation (6) iteratively, using the effective index method to calculate the effective index of the fundamental mode, the wavelength shift due to a refractive index perturbation of $1 \times 10^{-3}$ RIU was estimated. This wavelength shift was divided by the refractive index perturbation to obtain the theoretical sensitivity of this microresonator. The theoretical TE and TM sensitivities were 20 nm/RIU and 25 nm/RIU, respectively. Considering the measured sensitivity of 26 nm/RIU, the relative error of the prediction for the TE and TM modes is found to be 23% and 3.9%, respectively. The TM fundamental mode’s predicted sensitivity most closely matches the measured sensitivity. On this basis, the measured
mode was most likely the TM fundamental mode, but further experiments would be necessary to confirm that the polarization was TM.

6.2 EBL Process Development Microresonators

6.2.1 Transmission Curve Fitting Method

The purpose of this analysis is to show the trend of the coupling parameter $t$ with the interlayer dielectric thickness and to estimate the internal loss of the microresonators using both the symmetric ($t_1 = t_2$) coupling model and the asymmetric coupling model ($t_1 \neq t_2$). Using the equations described in section 2.5.1 along with an algorithm for fitting transmission model curves to measured spectra (see Appendix D: Microresonator Transmission Function Modeling Method), the round-trip loss coefficient, $a$, and the coupling parameters, $t_1$ and $t_2$, were extracted from measured spectra. The internal loss, $(\alpha)$ of the tested microresonators, which includes the material, bending, and scattering loss, was calculated from $a$.

6.2.2 Internal Loss and Bus-to-Resonator Coupling

To extract the coupling and loss parameters for the microresonator, both the symmetric and the asymmetric coupling models were applied. The results of fitting the symmetric coupling model to the measured data are shown in Figure 47, Figure 48, and Figure 49. The figures include a plot of the drop port spectrum adjusted to compensate for the longer optical path length from input port to drop port relative to the optical path.
length from input port to throughput port, using the estimated bus waveguide propagation loss of 6.8 dB/cm.
Figure 47. Measured spectra and transmission curve fits overlaid with the measured spectra scaled using the fit parameters for EBL_Test1 (900 nm interlayer dielectric). (a) Measured spectra. (b) Symmetric model. (c) Asymmetric model.
Figure 48. Measured spectra and transmission curve fits overlaid with the measured spectra scaled using the fit parameters for EBL_Test2 (1200 nm interlayer dielectric). (a) Measured spectra. (b) Symmetric model. (c) Asymmetric model.
Figure 49. Measured spectra and transmission curve fits overlaid with the measured spectra scaled using the fit parameters for EBL_Test3 (1400 nm interlayer dielectric). (a) Measured spectra. (b) Symmetric model. (c) Asymmetric model.
While the asymmetric model fit well to both the throughput port and the drop port spectra, the significance of the calculated results with the asymmetric solutions was insufficient to actually make any strong conclusions about coupling asymmetry. Specifically, the error in the analysis for the asymmetric model, due to a combination of the unknown error in the propagation loss estimate and the relative output coupling coefficients for the drop port and the throughput port, provided a level of uncertainty that precluded justification of conclusions based on the asymmetric model calculations. In order to perform an analysis of asymmetric coupling, these errors would need to be better understood.

Because the symmetric model does not require as many assumptions to be made as the asymmetric model does, it is a better model when the errors in those assumptions is unknown. The symmetric model also becomes a good approximation for the more general asymmetric model for highly decoupled microresonators \((t_1 \approx t_2 \approx 1)\) [122]. For a waveguide coupled microresonator that is not highly decoupled from its bus waveguides, the symmetric model cannot provide an accurate estimate of the round-trip loss coefficient \((a)\) or the coupling parameter for the drop coupler \((t_2)\), unless it is known \(a\) priori that the coupling is symmetric. However, both the asymmetric model and the symmetric model provide the same result for the coupling parameter for the throughput coupler \((t_1)\) under any condition. The reason that \(t_1\) is the same for both models is because in both models, \(t_1\) is a function of only the extinction ratio at the throughput
port and the Z factor, which is a function of the Q factor of the waveguide coupled microresonator (see equations (37) and (51)).

For these reasons, the analysis of the EBL test devices is limited to utilizing the symmetric model to estimate the coupling parameter for the throughput coupler, \( t_1 \), for the three EBL test devices. For EBL_Test3, the coupler parameters \( t_1 \) and \( t_2 \) are close enough to 1 that the symmetric model provides a reasonable estimate for both \( t_2 \) (\( t_2 \approx t_1 = t \)) and for the round-trip loss coefficient, \( a \). The internal loss \( \alpha \) is calculated from \( a \) to provide an estimate of the loss in units of dB/cm. For EBL_Test1, EBL_Test2, and EBL_Test3, \( t_1 \) was estimated to be 0.525, 0.823, and 0.953 by fitting the symmetric coupling model to the measured throughput port spectrum. The throughput coupler self-coupling parameter (\( t_i \)) was calculated to be 0.525, 0.823, and 0.953 for EBL_Test1, EBL_Test2, and EBL_Test3, respectively. The self-coupling parameter varies as expected, increasing with the increase in the interlayer dielectric thickness, or the gap between the throughput waveguide and the microresonator, for each of the test devices. The gaps for the test devices were 900 nm for EBL_Test1, 1,200 nm for EBL_Test2, and 1,400 nm for EBL_Test3. The internal loss \( \alpha \) for EBL_Test3 was estimated to be 13.1 dB/cm. The simulated internal loss for EBL_Test3 was 16.8 dB/cm for the TE fundamental mode and 13.4 dB/cm for the TM fundamental mode, and includes the combined effects of the material, bending, and surface scattering losses. These loss mechanisms were found to be the primary source of loss in planar microresonator
cavities in previous work [122, 124]. The error between the theoretical internal loss and the measured internal loss is about 28% for the TE mode and about 2.3% for the TM mode. Because the predicted internal loss overestimates the measured internal loss, it is likely that all of the important loss factors were considered. A better measurement of the surface scattering loss parameters, specifically that of the surface roughness of the vertical sidewalls and the roughness autocorrelation length on all surfaces, would likely improve the agreement between the theory and experiment. Also, determination of the polarization of the mode utilized in the sensor measurements would be necessary to confirm the agreement between the theoretical internal loss and the measured loss.

### 6.2.3 Sensitivity

The sensitivity of two of the EBL test devices was measured using the swapping technique with 2% D-glucose. The predicted sensitivity from the sensitivity design curves (Figure 13) for the TE and TM modes, considering a microresonator waveguide width of 1.5 μm and height of 1.9 μm, was 85 nm/RIU and 64 nm/RIU for the EBL test devices. The measured sensitivities for EBL_Test1 and EBL_Test3 were 84 nm/RIU and 82 nm/RIU, respectively. The error in the estimates for both the TE and the TM modes were 1.2% and 24%, respectively, for EBL_Test1 and 3.7% and 22%, respectively, for EBL_Test3. These results suggest that the TE mode was likely the measured polarization. However, determination of the measured polarization would be necessary to confirm the agreement between theory and experiment.
6.3 **EBL Defined Top Plate Embedded Microresonator**

6.3.1 **Internal Loss and Bus-to-Resonator Coupling**

Three EBL patterned top plate-embedded microresonator devices were fabricated on Pyrex substrates and tested. The spectrum was measured at the drop port and the throughput port for the device with the highest $Q$ factor of the three (EBL_TopPlate1). The coupling and loss parameters for this device were extracted from the throughput spectrum using the symmetric coupling model for the reasons given in section 6.2.2. The measured spectra correspond to that of the fundamental TM mode of the microresonator (see section 5.3.3.3). The measured spectra for EBL_TopPlate1 and symmetric model fits to the spectra are shown in Figure 50.
Figure 50. Measured spectra and transmission curve fits overlaid with the measured spectra scaled using the fit parameters for EBL_TopPlate1 (1400 nm interlayer dielectric). (a) Measured spectra. (b) Symmetric model.

From the model fits, the internal loss of EBL_TopPlate1 was calculated to be 20.0 dB/cm and the self-coupling parameter ($t$) of EBL_TopPlate1 was calculated to be 0.889. Using the surface scattering parameters measured from EBL_Test3 and the material and bending loss expected for a microresonator waveguide with dimensions of 1.2 μm width and 2 μm height, the internal loss of the microresonator was estimated to be 12.5 dB/cm
for the TM mode. These results indicate that EBL_TopPlate1 was affected both by more internal loss (the sum of $\alpha_{\text{mat}}$, $\alpha_{\text{bend}}$, and $\alpha_{\text{ss}}$ was greater) and by more coupling loss ($\alpha_{\text{coup}}$ was greater) than the comparable EBL test device (EBL_Test3) with the same interlayer dielectric thickness. The discrepancy might be due to increased surface roughness relative to the EBL_Test3 device and/or higher bending loss than expected. For these reasons, a lower Q factor (8,400) was measured for EBL_TopPlate1 than for EBL_Test3.

6.3.2 Sensitivity

The measured sensitivity was compared to the predicted sensitivity for the TM mode, which was identified as the only polarization that produced observable resonances in the drop port of EBL_TopPlate1 (see section 5.3.3.3). The predicted sensitivity for the TE and the TM fundamental modes was 130 nm/RIU and 88 nm/RIU, respectively, and the measured sensitivity for the TM mode was 89 nm/RIU (TM). The relative error of the predicted sensitivity is, therefore, 2.2%. Such a close agreement between the theoretical result and the experimental result indicates that the theoretical model utilized to predict the sensitivity as a function of waveguide width and height is sound.
7. Conclusions

Towards the goal of developing an integrated biosensing system, the primary result of this work was the development of a new integrated sensor design and fabrication process was developed to embed a microresonator sensor in the top plate of a microfluidic device and the successful demonstration of that device. This demonstration represents the first truly embedded sensor in an electrowetting-on-dielectric (EWD) microfluidic system in which the full functionality of the sensor and the microfluidic system were maintained. A brief list of the highlights of this work includes,

1. Measurement of the highest reported FOM for an SU-8 microresonator at 1550 nm.
2. First demonstration of a top plate embedded microresonator with an EWD system.
3. Addressing the sensor with a single droplet and demonstration of the ability to move droplets onto and off of the sensor and perform sensing.
5. An advanced design and testing methodology for microresonator sensors, based on recent theoretical developments and on numerical methods applicable to 2-D optical (channel) waveguides, was presented in this thesis.
6. Prediction of an improvement in FOM, using the design methodology described in this thesis, by adjusting the microresonator waveguide dimensions and validation of that prediction.

Integration of the optical system with the electrowetting-on-dielectric (EWD) system was achieved, but with some loss of functionality in the EWD microfluidics. The sensor was able to sense glucose in the EWD microfluidic system, while at the same time, the insertion loss of the optical system was negligibly affected by the EWD system and the EWD system functionality was unaffected, except at the sensor location. At the sensor location, the ability to actuate droplets on-and-off was maintained, but only for a limited number of cycles. A reasonable hypothesis for the limited cycle number is that the absence of ITO from a large portion of the droplet contact area of the top plate affects the electrical conductivity in that contact, which adversely affects the droplet actuation capability. In future work, the effect of the size of the etched ITO feature relative to the droplet’s contact area on the ability to actuate droplets under such a feature could be examined.

Biosensing with polymer microresonators made with SU-8, or with other polymers, is viable. The potential of polymer microresonator sensors remains mostly untapped at this point, primarily due to the need for careful design of the optical system and the microresonator waveguide to maximize the sensitivity and Q factor and to maintain a low insertion loss. The optimal design of these sensors will enable these
sensors to directly detect important signals for biosensing, such as the hybridization of DNA, while leveraging the added benefits of using polymer materials, such as the ability to directly immobilize probe molecules to the polymer surface for specific biomolecule detection, the compatibility of polymer materials for device integration, and the low cost of polymer materials. By modeling the sensitivity of the device, it was found that tall and thin waveguides had the highest sensitivities. This shape of waveguide was also found to correspond with the highest predicted figures of merit when material and bending losses were considered. Additionally, surface scattering loss was shown to be an important contribution to the microresonator propagation loss. Because of the importance of surface scattering to the loss in the microresonator, careful selection and control of fabrication techniques are necessary to minimize the surface roughness and to approach the material and bending loss limited figures of merit. For example, direct writing of the microresonator waveguide by EBL patterning might be inferior to photolithographic patterning in terms of the resulting surface roughness of the fabricated waveguide, because of the more pixellated nature of the e-beam exposure. These results will help to guide future improvements of the sensor performance.

7.1 Future Work

7.1.1 Investigate EWD Ground Plane Patterning Effects

Investigation of the effect of varying the shape and the ratio of the characteristic length/area of an etched region in the EWD ground plane to the characteristic
length/area of an EWD electrode and the alignment of these two regions on the ability to actuate droplets onto and off of the electrode below the etched ground plane feature (sensor electrode) is necessary to characterize and fully understand the trade-off between sensor integration in the top plate and the functionality of EWD at the sensor location. With an understanding of this trade-off, it would be possible to design the system both to optimize sensor performance and droplet actuation performance at the sensor electrode. Additionally, the effect of interdigitated electrodes on the actuation of droplets under an etched ground plane region should be investigated. There was some indication during testing that interdigitated electrodes performed better at the sensor electrode. No cycle dependence was observed with an interdigitated electrode EWD system, but the cycles were spaced rather far apart in time compared to the cycles done for the glucose measurement.

7.1.2 Integrate an MSM PD with a Top Plate Embedded Vertically-Coupled Microresonator Sensor

Theoretically 98% output coupling efficiency could be achieved with an MSM PD integrated into the top plate embedded microresonator optical system [168]. The approach would be to apply the photodetector (PD) to the substrate prior to coating the substrate with the PECVD SiO$_2$ layer for embedding the waveguides. The bus waveguides would be aligned with the PD and would overlap it.
7.1.3 Robust Validation of the Design Methodology and Improvement of the Waveguide Coupling Model

While the design and characterization methods presented in this thesis cover the primary design elements for the microresonator sensor, and some of the initial results show good agreement between theory and experiment, a more robust validation is necessary to confirm the accuracy of the design methodology. The first step would be to accurately measure the complex refractive index of all of the materials with variable properties that are used to make the waveguides (PECVD SiO$_2$, SU-8, Cytop). Additionally, accurate surface roughness characterization of the microresonator waveguide surfaces, including the sidewalls as well as the top and bottom surfaces, would be necessary. Designing the microresonator waveguide to be symmetrically coupled to its bus waveguides is very important, because the ability to utilize the symmetric model greatly simplifies the measurement of the coupling and loss parameters of test devices. Using very accurately measured parameters as inputs to the models should improve the agreement between theory and experiment for the sensitivity and the internal loss of the microresonator. Additionally, and most importantly, the more accurate input parameters might resolve the significant discrepancy between the theoretically predicted and the measured coupling efficiency between the bus waveguide and the microresonator. If after this careful analysis the model predictions continue to show a significant systematic discrepancy with the experimentally measured parameters, then other effects would have to be considered.
One of the most important effects that could alter the coupling efficiency as well as the sensitivity and Q factor of the microresonator is the absorption of water by the SU-8. The absorption of water lowers the refractive index of the SU-8 polymer. The refractive index of SU-8 can also be altered by stress [109]. Significant built-in stress might be present in the fabricated SU-8 waveguides, because of the difference in the coefficient of thermal expansion (CTE) between SU-8 and SiO₂.

### 7.1.4 Test DNA Sensing with an Optimized Sensor

Initial results with DNA sensing were promising (see Appendix E: DNA Sensing Experiments). The sensor that was used to detect DNA hybridization failed to detect a signal. There were two problems that could have caused this negative result:

1. Low sensor sensitivity.
2. Low hybridized DNA density.

The low sensitivity of the photolithographically patterned sensors was not apparent at the time, because the techniques utilized to estimate sensitivity yielded inflated values. Thus, the sensitivity was as good as unknown, along with the efficiency of the DNA hybridization. With the rapid and accurate sensitivity characterization method presented in this thesis, an accurate estimate of the sensor sensitivity can now be made. With the ability to design and accurately characterize the sensor, a sensor capable of detecting DNA, having comparable performance to those that have already been
demonstrated to detect DNA, can be designed and fabricated. This capability addresses the first problem, that of low sensitivity.

With the first problem resolved, the focus can then be turned to the second possible problem, low hybridized DNA density. If a DNA hybridization signal cannot be measured by simply repeating the DNA hybridization experiments as they were performed previously on the microresonator sensor, characterization of the hybridized DNA density and/or the probe DNA density might be necessary to explain the null result and to develop a better DNA probe immobilization or DNA hybridization protocol. The density of hybridized target DNA and probe DNA on the sensor surface can be estimated by measuring these quantities on test spots on a thin film of SU-8. Test spots were used previously (see Figure 56) to qualitatively characterize probe DNA immobilization and target DNA hybridization. One way to measure the hybridized DNA density would be to compare the fluorescence of Cy3 fluorophore-tagged target DNA hybridized to probe DNA with the fluorescence of spots of Cy3 on a fluorescence calibration slide (Scanner Calibration Slide, Full Moon Biosystems), which have known surface densities of Cy3 fluorophore. A more accurate method, which is probably more difficult method to implement, is to utilize \(^{32}\text{P}\) radiolabeled target DNA as per the density measurement technique reported by NIST [169].

In addition to experimental methods, theoretical simulations can also be utilized to gain insight into the DNA hybridization signal. A theoretical model of the DNA
hybridization signal could be implemented to predict the expected signal of DNA hybridization. This model would have to include the refractive index versus density of the DNA film for the unhybridized and the hybridized case, and possibly the thickness of the film before and after hybridization. Because a film of DNA 20-30 mers in length has a physical length of around 10-20 nm, very high resolution simulations would be needed to obtain accurate estimates of the guided mode phase constant before and after DNA hybridization. The model described in this thesis for estimating sensitivity, combined with a suitable method for calculating the required phase constants from the waveguide’s refractive index profile, could be utilized to estimate the expected wavelength shift due to DNA hybridization.
Appenndix A: Transmission Analysis for an N-Waveguide Coupled Microresonator

Because the form of the equations (14)-(24) remain the same for any throughput port \(i\) for any number of coupled waveguides and (28)-(30) and (32) for any drop port \(j\) for any number of coupled waveguides (with one additional term in the numerator), these equations can be generalized for the case of \(N\) coupled waveguides. The relevant equations for all spectral measurements in the generalized model are

\[
\Delta \lambda_{FWHM} = \left( \frac{\Delta \lambda_{FSR}}{\pi} \right) \arccos \left( 2 - \frac{1}{2Z} - \frac{Z}{2} \right) \text{ and} \tag{70}
\]

\[
Z = a \prod_{k=1}^{N} t_k = 2 - \left( \cos^2 \left( \frac{\pi}{F} \right) - 4 \cos \left( \frac{\pi}{F} \right) + 3 \right)^{1/2} - \cos \left( \frac{\pi}{F} \right). \tag{71}
\]

Further generalizing these equations for use with peak width measured at any fraction \(X\) of the resonance peak height (\(X\) is defined for nulls by (20)) yields

\[
\Delta \lambda_{FWHM} = \left( \frac{\Delta \lambda_{FSR}}{\pi} \right) \arccos \left( X + 2Z + XZ^2 - Z^2 - 1 \right) \text{ and} \tag{72}
\]

\[
X^{-1/2} \left[ X \cos^2 \left( \frac{\pi \Delta \lambda_{FWHM}}{\Delta \lambda_{FSR}} \right) - 2 \cos \left( \frac{\pi \Delta \lambda_{FWHM}}{\Delta \lambda_{FSR}} \right) - X + 2 \right]^{1/2} + X \cos \left( \frac{\pi \Delta \lambda_{FWHM}}{\Delta \lambda_{FSR}} \right) - 1 \tag{73}
\]

The generalized expression for \(\phi\) with \(N\) couplers is given by

\[
\phi = \beta_{n0}(\lambda)L + \sum_{i}^{N} \phi_i = \frac{2\pi n_{eff,\text{ms}}(\lambda)L}{\lambda} + \sum_{i}^{N} \phi_i = 2\pi \left[ \frac{\Delta \lambda_{FWHM}}{\Delta \lambda_{FSR}} + 1 \right] \tag{74}
\]
where $i$ is the $i$-th coupler. Couplers are numbered by integers in increasing order in the direction of propagation in the microresonator. Generalized equations for the throughput port transmission spectrum are

$$
{t'}_i = \left[ \frac{E_{2,i}}{E_{1,i}} \right]^2 = \frac{1}{t_i^2} \frac{t_i^4 + Z^2 - 2t_i^2Z \cos(\phi)}{1 + Z^2 - 2Z \cos(\phi)},
$$

(75)

$$
{t'}_{T_i} = \frac{1}{t_i^2} \frac{(t_i^2 - Z)^2}{(Z - 1)^2},
$$

(76)

$$
{t'}_{ER_i} = \frac{(t_i^2 + Z)^2(Z - 1)^2}{(t_i^2 - Z)^2(Z + 1)^2},
$$

(77)

$$
{t'}_i = \left( \frac{{t'}_{ER_i}^{1/2} Z \mp Z^2 + {t'}_{ER_i}^{1/2} Z^2}{d \cdot \mp Z + {t'}_{ER_i}^{1/2} \pm 1} \right)^{1/2}.
$$

(78)

Generalized equations for the drop port are

$$
{d T}_{ij} = \left[ \frac{E_{2,j}}{E_{1,j}} \right]^2 = \left( \frac{Z^2}{\prod_{k=1}^{N} t_k^2} \right)^{FD} \frac{(t_i^2 - 1)^2 \prod_{k=i+1}^{j-1} t_k^2(t_j^2 - 1)^2}{1 + Z^2 - 2Z \cos(\phi)},
$$

(79)

$$
{d T}_{ij} = \left( \frac{Z^2}{\prod_{k=1}^{N} t_k^2} \right)^{FD} \frac{(t_i^2 - 1)^2 \prod_{k=i+1}^{j-1} t_k^2(t_j^2 - 1)^2}{(Z - 1)^2},
$$

(80)

$$
{d ER}_i = \frac{(Z + 1)^2}{(Z - 1)^2},
$$

(81)

$$
Z = \frac{d \cdot ER_i^{1/2} - 1}{d \cdot ER_i^{1/2} + 1}.
$$

(82)
where $j$ is the $j$-th drop bus waveguide and $i \neq j$. The term $\prod_{k=i+1}^{j-1} t_k^2$ in (79) and (80) arises when considering the three waveguide coupled case. For a system with $N$ couplers, generally $N$ spectral measurements are required to measure all of the coupling parameters. Less measurements are required if some of the couplers are designed to have equal coupling parameters.
Appendix B: Application of the Variational Theory to Microresonator Sensors

Using the variational theory of waveguides, equation (55) can be further simplified by deriving expressions for the term $\delta n_{\text{eff}}/\delta n_s$ in terms of the overlap of the field intensity with the sensing region. This simplification provides useful insights into the microresonator sensitivity. A variational expression for the phase constant of a guided mode traveling in the $z$-direction in terms of the vector fields is given by [170]

$$\beta = \frac{\iint_{s} dxdy \left( \omega \mathbf{E}^* \cdot \mathbf{E} + \omega \mu_0 \mathbf{H}^* \cdot \mathbf{H} + \left[ \mathbf{E}^* \cdot \nabla \times \mathbf{H} - \mathbf{H}^* \nabla \times \mathbf{E} \right] \right)}{\iint_{s} dxdy (\mathbf{E} \times \mathbf{H}^* + \mathbf{E}^* \times \mathbf{H}) \cdot \hat{z}}$$

(83)

where $\beta$ is the phase constant of the guided mode, $\mathbf{E}$ and $\mathbf{H}$ are the vector electric and magnetic fields, $\omega$ is the angular velocity of the electromagnetic wave, and $\mu_0$ is the permeability of free space. Using this variational expression, a relatively simple equation for the change in the phase constant due to a perturbation of the permittivity in the sensing region of a waveguide, $\Delta \varepsilon_s$, can be derived as [170]

$$\Delta \beta = \omega \frac{\iint_{s} dxdy \Delta \varepsilon_s \mathbf{E}^* \cdot \mathbf{E}}{\iint_{s} dxdy (\mathbf{E} \times \mathbf{H}^* + \mathbf{E}^* \times \mathbf{H}) \cdot \hat{z}}$$

(84)

where the $s$ denotes integration over the cross-sectional area that comprises the sensing region. In order to derive (84), it is assumed that the perturbation in the permittivity is small enough that the difference between the perturbed field profiles and the unperturbed field profiles is negligible [171, 172]. After some manipulation and by writing the finite differences as infinitesimal differences, (84) can be rewritten as
\[
\frac{\partial n_{\text{eff}}}{\partial n_s} = \frac{c}{n_s} \frac{\iint \! \! \! \! \iint \! \! \! \! \iint dxdy \varepsilon_s E^* \cdot E}{\iint \! \! \! \! \iint dxdy \text{Re}\{E \times H^*\} \cdot \hat{z}},
\]

(85)

where the denominator was simplified by utilizing a more condensed expression for the
time average power density for an electromagnetic wave traveling in the z direction
[173]. Utilizing the relationship between the rate of power flow of the guided mode and
the total electromagnetic energy per unit length, which, without any simplifications, is
written as [173]

\[
\frac{1}{2} \iint \! \! \! \! \iint \! \! \! \! \iint dx dy \text{Re}\{E \times H^*\} \cdot \hat{z} = \frac{v_g}{2} \iint \! \! \! \! \iint dx dy \left(\varepsilon E \cdot E^* + \mu H \cdot H^* \right),
\]

(86)

where \(v_g\) is the group velocity, and using the relationship \(v_g = c/n_g\), where \(n_g\) is the group
refractive index, equation (85) can be rewritten as

\[
\frac{\partial n_{\text{eff}}}{\partial n_s} = \frac{n_g}{n_s} \frac{\iint \! \! \! \! \iint \! \! \! \! \iint dxdy \varepsilon_s E^* \cdot E}{\iint \! \! \! \! \iint dxdy \left(\varepsilon E \cdot E^* + \mu H \cdot H^* \right)}.
\]

(87)

Because the numerator of (87) represents the electric field energy per unit length in the
sensing region and the denominator represents the total electromagnetic field energy per
unit length for the entire waveguide cross-section, it is possible to conclude that the
sensitivity of a waveguide sensor to a change in the permittivity of the sensing region is
positively correlated with the overlap of the electric field intensity with the sensing region. Also,
it can be concluded that the sensitivity is proportional to the electric field energy in the
sensing region as a fraction of the total electromagnetic energy in the waveguide.
Many waveguide sensor researchers utilize a result that asserts that $\frac{\delta n_{\text{eff}}}{\delta n_s} \approx \Gamma_s$, where $\Gamma_s$ is the fraction of the total field intensity (including electric and magnetic field components) in the sensing region [65, 94, 174]. $\Gamma_s$ will hereafter be referred to as the overlap factor. For an electromagnetic wave traveling in the z direction, $\Gamma_s$ is defined as [175]

$$\Gamma_s = \frac{\iint_{S} dx dy \langle |\mathbf{S}| \rangle \cdot \hat{z}}{\iint_{S} dx dy \langle |\mathbf{S}| \rangle \cdot \hat{z}}$$  \hspace{1cm} (88)$$

where $\mathbf{S}$ is the Poynting vector. The expectation value of the magnitude of $\mathbf{S}$, which is the average rate of energy flow, is given by [173]

$$\langle |\mathbf{S}| \rangle \cdot \hat{z} = \frac{1}{2} \iint_{S} dx dy \text{Re}\left(\mathbf{E} \times \mathbf{H}^*\right) \cdot \hat{z}.$$  \hspace{1cm} (89)$$

Using the field relations for TE and TM modes of a slab waveguide, given in [115], the overlap factors are found to be

$$\Gamma_{s,\text{TE}} = \frac{\iint_{S} dx dy \left|E_x\right|^2}{\iint_{S} dx dy \left|E_x\right|^2} \text{ and}$$  \hspace{1cm} (90)$$

$$\Gamma_{s,\text{TM}} = \frac{\iint_{S} dx dy \frac{1}{\varepsilon} \left|H_x\right|^2}{\iint_{S} dx dy \frac{1}{\varepsilon} \left|H_x\right|^2}$$  \hspace{1cm} (91)$$

for the TE and TM modes, respectively. The $1/\varepsilon$ term must remain in the integrand for the TM mode fields, because it is generally only piecewise continuous for dielectric waveguides. Using equations (85), (90), and (91), and the appropriate Maxwell’s
equations relating \( \mathbf{E} \) and \( \mathbf{H} \) from [173], solutions for \( \delta n_{\text{eff}}/\delta n_s \) for TE and TM modes can be derived as

\[
\frac{\partial n_{\text{eff}}}{\partial n_s} = \frac{n_s}{n_{\text{eff}}} \Gamma_{s,TE} \quad \text{and} \quad (92)
\]

\[
\frac{\partial n_{\text{eff}}}{\partial n_s} = \frac{n_s}{n_{\text{eff}}} \left[ 2 \left( \frac{n_{\text{eff}}}{n_s} \right)^2 - 1 \right] \Gamma_{s,TM} , \quad (93)
\]

respectively. These equations for the sensitivity of the TE and TM modes of a slab waveguide exactly match equations derived using a different approach [131]. If the differences between the core and cladding refractive indices of the slab waveguide are small, \( n_{\text{co}} - n_{\text{cl}} \ll n_{\text{cl}} \) (i.e. a weakly guiding waveguide), the additional approximation that \( n_s/n_{\text{eff}} \approx 1 \) can be made [176]. Therefore, a general approximation to \( \delta n_{\text{eff}}/\delta n_s \) for weakly guiding waveguides is

\[
\frac{\partial n_{\text{eff}}}{\partial n_s} \approx \Gamma_s . \quad (94)
\]

This approximation is often utilized to estimate the optical gain in semiconductor lasers [175, 176]. The foregoing analysis provides the basis of the approximation for \( \delta n_{\text{eff}}/\delta n_s \) given by equation (94) used by many authors to estimate waveguide or microresonator sensitivity. A notable example of the use of the approximation in (94) was published by Zhu, et al. [174], and was found to be accurate within a few percent of a more rigorous three-layer Mie scattering model for estimating the sensitivity of a liquid core ring resonator.
Appendix C: Analysis of Sensor Drift Behavior

**Experiment Methodology**

To expose the sensor to water for a long period of time, a cylindrical glass gasket, which could hold up to about 225 µL of fluid, was used to confine a volume of water on top of the sensor. Evaporation was prevented by covering the top of the cylinder with a small piece of Parylene-M film after filling the cylinder to the top with water. The glass cylinder was bonded to the device surface using EP 1121 epoxy. The glass cylinders, the EP 1121 epoxy, and the Parylene-M film were graciously provided by Dr. Scott Wolter. This test was performed with a photolithographically patterned test device. Application of the gasket did not affect insertion loss appreciably.
Several experiments were performed to assess the drift of the resonant wavelengths with the sensor immersed in water. In all experiments, the gasket was filled near to the top before covering it with the Parylene-M film. Immediately after adding water to the gasket and covering it with Parylene-M, spectra were recorded at ~12 s intervals for 3-5 hours. After completing an experiment, the gaskets were emptied, dried with a stream of N2 gas, and left exposed to the laboratory environment for at least 12 hours prior to the next experiment. The 12 hour desorption time was considered ample time for complete desorption of water from the SU-8, because the observed resonant wavelength drift of a test microresonator that had been immersed in water for 36 hours due to water desorption was almost gone after 12 hours after the water had been removed (see Figure 52).
Figure 52. Desorption of water from a microresonator that had been immersed in water for 36 hours.

**Drift Consistency Between Devices Immersed in Water**

To explore the drift behavior between different devices, the sensor drift was measured twice for a set of three photolithographically patterned test sensors. The plots in Figure 53 show sensorgrams of the sensor drift for the three test sensors for two consecutive drift measurements.
Figure 53. Sensor response of 3 similar microresonators (fabricated in the same batch) due to absorption of water. (a) Test 1. (b) Test 2.
The results show that the drift behavior was similar for the three devices for both consecutive drift measurements, that the drift was nonlinear, and that the magnitude of the drift was different for each sensor. Also, the asymptotic behavior of the drift changed between the first test and the second test.

The drift of positive slope observed in the first drift measurement for all three of the test devices was most likely caused by the incorporation of water into the pores of the PECVD SiO$_2$ under the microresonator. It is known that SiO$_2$ deposited by low temperature processes is porous and is very reactive with water for a short period of time post-deposition [177]. The porosity of the PECVD SiO$_2$ films in devices fabricated for this thesis was qualitatively assessed by etching a film of PECVD SiO$_2$ deposited on a film of thermally grown SiO$_2$ with buffered-oxide etchant for 3 minutes. The SEM image of the result is shown in Figure 54.
Figure 54. SEM image of a 2 μm film of PECVD SiO₂ on top of a 4 μm film of thermally grown SiO₂ on a silicon wafer etched in buffered oxide etchant for 3 minutes. The channel in the PECVD SiO₂ was for the bus waveguide of a photolithographically patterned test microresonator device.

In the SEM image, the PECVD SiO₂ film appears significantly more porous than the thermally grown SiO₂ film. Over time, the dry pores of a PECVD SiO₂ film fill up with water from the humid environment outside of the PECVD deposition chamber [177]. The incorporation of water into the pores of the PECVD SiO₂ film increases its refractive index, because water, which has a refractive index of 1.318, fills voids that were previously occupied by air, which has a refractive index of 1. According to equation (59), a positive shift in the wavelength would result from an increase in the substrate refractive index. Therefore, it is likely that applying water to the surface of the PECVD SiO₂ accelerates the process of incorporation of water into the film and that this
incorporation is manifest in the sensorgram as a positive shift in the resonant wavelength. Additionally, the reaction of a freshly deposited PECVD SiO$_2$ film, which is highly reactive with water [177], might contribute to a positive change in its refractive index.

On the second run, the asymptotic behavior of the drift is negative. This drift mode is the main drift mode observed for all of the devices tested for this thesis, except for those tested within about 1 week after fabrication. This drift mode is probably caused by the reduction in refractive index of the SU-8 polymer material with which the microresonator was fabricated, due to the tendency of SU-8 to absorb water. The absorption of water by SU-8 is known to be consistent with the Fick model of diffusion and has a diffusion constant, $D$, of $1.25\times10^{-9}$ cm$^2$/s [178, 179]. Also, SU-8 has been found to swell significantly when immersed in water [178].

There are five physical phenomena that are likely to occur from water absorption that could cause the resonant wavelengths of the SU-8 microresonator cavity to shift:

1. Incorporation of water into the polymer matrix.
2. A reduction in the density of the polymer due to swelling.
3. A change in the cavity length.
4. Delamination of the cavity from the substrate.
5. Irreversible chemical reactions of the polymer with water.
Incorporation of water into the polymer matrix would reduce the refractive index of the polymer, because the refractive index of water is lower than the refractive index of SU-8. The result would be a blue shift of the resonant wavelengths. The swelling of the polymer would reduce its density and, therefore, its refractive index, which would result in a blue shift of the resonant wavelengths. An increase in cavity length would cause the resonant wavelengths to red shift, according to the resonance condition, given by equation (5). Slight delamination of the cavity would allow water, which has a lower refractive index than the SiO₂ substrate, to move into spaces beneath the microresonator waveguide. Thus, delamination would effectively lower the substrate refractive index, causing the resonant wavelengths to blue shift. Finally, irreversible chemical reactions with water could effect the drift, but it is not clear if these would result in a red shift or a blue shift. Except when monitored during the first immersion in water and within about 1 week of fabrication, sensor drift was always observed to be a blue shift (negative slope in the sensorgram). Therefore, phenomena that cause a blue shift (#1, 2, 4, and possibly 5) dominate the sensor drift.

In some of the sensorgrams, an oscillatory signal was observed. The oscillation was due to a small amplitude, small FSR, Fabry-Perot resonance from the optical system (either the bus waveguides or the input and/or output optical fibers) overlaid on top of the microresonator spectral response. This Fabry-Perot resonance was usually suppressed with the coherence control modulation function of the tunable laser system.
(HP8164A Lightwave Measurement System with the HP81680A tunable laser), which was turned off during the sensor drift experiments.

The change in the refractive index of the SU-8 polymer upon absorption of water has implications on the microresonator design. A change in the refractive index of the microresonator waveguide due to water absorption is expected to affect both the sensitivity and the coupling of modes between the bus waveguides and the microresonator waveguide. Model refinements to account for water absorption are subtle and would require many experiments to determine precisely how the water absorption affects the refractive index of the SU-8. Because accurate modeling was not a major goal of the work done for this thesis, such experiments were not pursued and the models were not adjusted to account for the water absorption.

**Drift Consistency Between Successive Immersions in Water**

To explore the variation of the sensor drift between successive immersions in water, a single microresonator device was immersed in water for 3-5 hours six times over a 6 day period to determine if the drift function would vary after a number of long term immersions in water. Between each drift measurement, the sensor was set in air for at least 12 hours to allow the water absorbed into the SU-8 polymer to desorb. The sensorgrams for these experiments are shown in Figure 55.
An interesting trend was observed through successive immersions. Initially, the drift due to the absorption of water had relatively large slope. For every subsequent test, the slope at was reduced as well as the total wavelength shift. Significant reductions in the drift slope and in the total shift were no longer observed after the fifth test. From these experiments, it seems that irreversible changes might occur in the SU-8 material during exposure with water that lead to a change in the drift function with time. More experiments would be required to verify this hypothesis. Also, these results suggest a method for conditioning the sensor to reduce the slope and the magnitude of the resonant wavelength shift due to the drift.
**Drift Compensation**

Because the wavelength drift was always present in these devices and varied across devices and from run-to-run, drift was compensated for by including either a linear or a quadratic function to model the drift when estimating the wavelength shift caused by introducing analytes to the sensor.
Appendix D: Microresonator Transmission Function Modeling Method

Equation (1), which relates the input power to the output power by the insertion loss, was used to fit either the drop or the throughput transmission models to the measured data by least squares fitting. The fit equation was

\[ P_{\text{out}} = C_{\text{sc}} T_{\text{res}} \]

(95)

where \( C_{\text{sc}} \) is a scaling coefficient and is given by

\[ C_{\text{sc}} = T_{\text{in}} T_{\text{prop}} T_{\text{out}} P_{\text{in}} \]

(96)

The scaling factor is so named, because by dividing the measured spectrum by the scaling factor, the measured data will line up with the curve for the theoretical transmission spectrum for the microresonator, plotted on a scale from 0 to 1. In general, \( C_{\text{sc}} \) is different for the throughput port and the drop port, because \( T_{\text{prop}} \) and \( T_{\text{out}} \) are not generally the same for both ports. In the following discussion, \( C_{\text{sc,thru}} \) and \( C_{\text{sc,drop}} \) denote the scaling coefficient for the throughput port and the drop port, respectively.

A procedure was developed and implemented for fitting (95) to noisy measured data. For modeling with both the symmetric and the asymmetric models, the first step performed was a least squares fit of the throughput port symmetric coupling model, using equation (34) with \( t_1 = t_2 = t \) for \( T_{\text{res}} \), to the measured throughput port spectrum, using initial estimates for \( C_{\text{sc}}, a, t \), the center wavelength of one of the resonances, and the free spectral range (FSR). The model parameters from the throughput port fit were
used to calculate the extinction ratio (\(tER\)) at the throughput port using equation (36) and were also used as initial values for least squares fitting of the drop port symmetric coupling model to the measured drop port spectrum.

Before fitting a model to the drop port spectrum, the measured drop port spectrum was scaled as per equation (43) to adjust for the path length difference of the drop port and the throughput port using the design path length difference of 0.3059 cm and the estimated bus waveguide propagation loss of 6.8 dB/cm. The adjusted drop port spectrum estimated the spectrum that would be measured at the drop port if the propagation length for the drop port was equal to that of the throughput port (i.e. effectively \(T_{prop,drop} = T_{prop,thru}\)). \(C_{sc\ell}\) as calculated by equation (96) would then be the same for both the drop port and the throughput port measurements, under the assumptions that the output coupling efficiency at both the drop and the throughput ports were exactly equal and that the propagation loss estimate of 6.8 dB/cm was exact. If these assumptions were accurate and the microresonator were symmetrically coupled to the two bus waveguides, then both the measured drop port spectrum and the measured throughput spectrum would have equal scaling factors and, if scaled by those scaling factors, would line up with the symmetric coupling model curves for the drop transmission and the throughput transmission, respectively. Any residual difference between the scaling parameters \(C_{sc\ell,drop,adj}\) and \(C_{sc\ell,thru}\) calculated from least squares fitting
of the symmetric transmission models would indicate that the coupling was asymmetric
\((C_{sc\text{,drop,adj}})\) is defined as the adjusted drop spectrum scaling parameter).

After appropriately scaling the drop port measured spectrum, a least squares fit
of the drop port symmetric coupling model to the measured drop port spectrum was
performed. The drop port symmetric coupling model was fit to the measured drop port
spectrum using equation (29) for \(T_{res}\) with \(t_1 = t_2 = t\). Using the drop port fitted
parameters, the expected value of the FWHM of the resonances in the drop port
spectrum was calculated using equation (31) with \(t_1 = t_2 = t\). The FWHM and the FSR,
which was calculated by the drop port model fit, were used to calculate the finesse \((F)\) of
the waveguide-coupled microresonator. The finesse was used to calculate the \(Z\)
parameter of the waveguide-coupled microresonator with equation (33). Finally, the
calculated values of \(Z\) and \(t'ER\) were utilized to calculate the two solutions for the
throughput coupler coupling parameter \(t_1\) using equation (37).

The decision to use the FWHM and the FSR from the drop port model fit was
based on some observations of the fitting algorithm. Some parameters derived from the
model fits to spectra measured at each port that should be the same no matter which
port they are measured from, such as the FWHM, FSR, or \(Z\), were found to be close but
not exactly matching. The discrepancy was probably due to a combination of fitting
error and measurement error. In the case that these parameters do not match when
measured from different ports, the average of the two calculated values may be the most
unbiased estimate of these parameters. However, because all of the sensing experiments reported in this thesis utilized spectrum measurements at the drop port and the Q factor was measured in drop port spectra, the drop port parameters were most relevant in these analyses. Thus, the system parameters FWHM, FSR, Z, and F derived from the model curve fits to the drop port spectra, being the most relevant to the measured data in this thesis, were used as estimates of these system parameters.

For the symmetric coupling case, \( t_1 = t_2 = t \). Therefore, once \( t_1 \) was calculated, the coupling parameter \( t \) at both ports was known. Using \( t \), the internal loss factor was calculated using the calculated value of the \( Z \) parameter and the definition of \( Z \) for the symmetrically coupled dual waveguide case, \( Z = aP \). Finally, using the definition \( a = \exp(-\alpha L/2) \), where \( L \) is the length of the resonant cavity, the exponential loss coefficient \( \alpha \) of the cavity was estimated. Two solutions for \( t \), and, therefore, also for \( a \) and \( \alpha \), were obtained using this method. For all of the transmission spectra analyzed with this algorithm, one of the two solutions for \( t \) always produced a negative value for \( \alpha \), which was physically impossible as there was no gain in the microresonator.

For the asymmetric coupling case, \( t_1 \neq t_2 \). Therefore, additional calculations were necessary to determine the drop coupler coupling parameter \( t_2 \). \( t_2 \) was calculated from \( t_1 \) using equation (40). In order to use equation (40), the maximum microresonator transmission at the drop port to that of the throughput port must be calculated. Using the scaled drop port power, equation (43), which relates the ratio of the maximum
power measured at the drop port to the maximum power measured at the throughput port to the ratio of the maximum microresonator transmission at the drop port to that of the throughput port, can be rewritten as

$$\frac{d_{T}}{n_{T}} = \frac{d_{adj}}{r_{r}} \frac{P_{in}}{T_{in}} \frac{T_{prop,thru} T_{thru}}{T_{prop,drop,adj} T_{drop}} P_{in} = \frac{d_{adj}}{r_{r}} \frac{P}{T_{prop,thru}} \frac{C_{scl,thru}}{C_{scl,drop,adj}},$$

(97)

where $d_{adj}/P$ is the adjusted drop port power on resonance and $T_{prop,drop,adj}$ is the effective waveguide transmission term for the adjusted drop waveguide. If the propagation loss compensation for the drop port were accurate, then the ratio of the fitted scaling factors would be equal to one. Thus,

$$\frac{d_{T}}{n_{T}} = \frac{d_{adj}}{r_{r}} \frac{P}{P}.$$

(98)

However, if the coupling is asymmetric, the fitted symmetric transmission models would not account for the difference in $t_1$ and $t_2$ and, therefore, would yield fitted scaling parameters that were not equal, even with the propagation loss compensation. Equation (97) can be rewritten in terms of symmetric model functions and rearranged to obtain an accurate value for the ratio $\frac{d_{adj}}{nr}P$:

$$\frac{d_{adj}}{nr}P = \frac{d_{symm}}{r_{r}} T_{symm} C_{scl,drop,adj,symmfit} C_{scl,thru,symmfit},$$

(99)

where $C_{scl,thru,symmfit}$ is the scaling parameter calculated from the throughput symmetric transmission model fit, $C_{scl,drop,adj,symmfit}$ is the scaling parameter calculated from the drop symmetric transmission model fit, and the superscript ‘symm’ indicates the assumption
of \( t_1 = t_2 = t \) for the respective transmission model function. Using the equality expressed in equation (98), the ratio \( \frac{d_T}{nT} \) is determined from the result of (99). Finally, \( t_2 \) can be calculated from equation (40) using this ratio and the solutions already obtained for \( t_1 \).

Since there are, in general, two solutions to \( t_1 \), there are at least two solutions to \( t_2 \), each of which corresponds to a particular \( t_1 \) solution. In fact, there are actually two solutions to \( t_2 \) for each \( t_1 \) solution. However, for all of the measured data to which this algorithm was applied, only one of the two solutions was physically realistic (i.e. between 0 and 1). The two remaining sets of solutions for \( t_1 \) and \( t_2 \) are both physically valid, because both yield internal loss factors \( (a) \) between 0 and 1. Additional information is necessary to determine which set of coupling coefficients is correct.
Appendix E: DNA Sensing Experiments

Some results from experiments with DNA hybridization are briefly summarized in this appendix. The intent is to show the main results of the work done toward the development of a DNA sensor.

The protocol followed for DNA hybridization was based on a method published by Marie, et al. [180]. Several experiments were performed to optimize the DNA hybridization efficiency prior to attempting the process on the sensor. DNA immobilization and hybridization efficiency was qualitatively assessed by using DNA tagged with the Cy3 fluorescent molecule. Probe DNA tagged with Cy3 was used to assess the probe DNA immobilization efficacy on an SU-8 surface. To assess hybridization efficiency, target DNA, which had a sequence complementary to that of the probe DNA and which was tagged with Cy3 fluorophore, was hybridized with untagged probe DNA immobilized to the SU-8 surface. The results of several experiments are summarized in Figure 56.
<table>
<thead>
<tr>
<th>Conditions</th>
<th>1 μM Probe</th>
<th>4 μM Probe</th>
<th>7 μM Probe</th>
<th>10 μM Probe</th>
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<tr>
<td><strong>No Ethanolamine Treatment</strong></td>
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<tr>
<td>Control Cy3-Probe</td>
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<tr>
<td>Control Probe Hybridized with Cy3-Target</td>
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<td>Patterned Substrate Cy3-Probe</td>
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<td>Patterned Substrate Probe Hybridized with Cy3-Target</td>
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<td><strong>1 Hr Ethanolamine Treatment</strong></td>
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<td>Control Cy3-Probe</td>
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<td>Control Probe Hybridized with Cy3-Target</td>
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<td>Patterned Substrate Cy3-Probe</td>
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<td>Patterned Substrate Probe Hybridized with Cy3-Target</td>
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</table>

**Figure 56.** DNA hybridization on patterned SU-8 regions on top of a Teflon coated microscope slide. Images of Cy3 fluorescence were taken with the stereomicroscope at Duke’s Light Microscopy Core Facility (LMCF).

Because the brightness of the fluorescence from the hybridized DNA spots was nearly as bright as that from the probe only spots for many of the conditions tested, the hybridization protocol was considered to be sufficiently good to attempt on a sensor.

The DNA hybridization protocol required heating the sensor to 37°C. To heat the
microresonator sensor, a copper vacuum chuck bonded to a Peltier device as in [181] was fabricated (Figure 57). The copper components were fabricated by the Duke Physics Instrument Shop.

![Copper vacuum chuck bonded to a Peltier device.](image)

**Figure 57. Copper vacuum chuck bonded to a Peltier device.**

The vacuum chuck held the device under test firmly in place to maintain optical alignment and helped to ensure good thermal contact between the device and the temperature-controlled chuck. A temperature sensor was also embedded into the chuck.

Several DNA hybridization experiments were performed on a sensor to see if a signal could be observed. The process of performing the DNA hybridization is described only described briefly here. The DNA hybridization was performed with a photolithographically patterned test sensor with a glass cylinder gasket bonded on top with EP 1121 epoxy (see Figure 51). Untagged probe DNA was immobilized on the sensor prior to applying the glass cylinder gasket. During the experiment, the gasket was filled with buffer solution and allowed time to heat before adding target DNA solution. Parylene-M film was applied to the top of the gasket to limit evaporation.
After heating for at least an hour, a concentrated solution of complementary target DNA tagged with Cy3 fluorophore was spiked into the solution. After waiting at least a half an hour, the gasket was emptied and washed out with the appropriate washing solutions. Figure 58 shows an image of the Cy3 fluorescence from DNA hybridized with the probe DNA that was immobilized on the SU-8 surfaces of the device for which the best results were obtained.

![Figure 58](image)

**Figure 58.** Fluorescence image of Cy3 tagged complementary DNA target hybridized to DNA probe immobilized on a photolithographically patterned test SU-8 microring resonator sensor with a 1250 μm diameter.

The observation of fluorescence in areas in which the probe DNA was immobilized indicated successful DNA hybridization. Probe DNA was not immobilized on all of the SU-8. Regions of SU-8 that were not covered with DNA probe are visible in the images as the dark parts of the rectangular SU-8 patterns adjacent to the microring resonator.
The fluorescence image also indicates that the selectivity for DNA immobilization and hybridization on SU-8 surfaces relative to the substrate surfaces was excellent. Unfortunately, the sensorgrams from these experiments never showed a clear signal corresponding to the DNA hybridization. This might have been caused by low sensor sensitivity (the photolithographically patterned sensors had low sensitivity) or by poor hybridization efficiency (hybridization efficiency was never quantitatively measured).
References


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Biography

I was born in Fairfax, VA, USA in 1984 and lived in Bethesda, MD until 1988. From 1988-2002, I lived primarily in Lower Gwynedd and North Wales, PA, just north of Philadelphia, except for a two year period in which I lived in Cupertino, CA. During those years, I attended Blue Bell Elementary School, Wissahickon Middle School, Penn Charter in Pennsylvania. During the years that I was in California (1997-1999), I attended Kennedy Middle School and The Harker School. After graduating from Penn Charter in 2002, I attended the Pennsylvania State University in State College, PA. While at the Pennsylvania State University, I worked in the labs of Prof. Jian Xu and of Prof. Mohsen Kavehrad. In the spring of 2005, I attended Tohoku University in Sendai, Japan as a foreign exchange student in the Japanese Year Program in English. As part of the program, I worked in a research laboratory as a student of Prof. Keiichi Edamatsu at the Laboratory for Nanoelectronics and Spintronics. In 2006, I received the bachelor of science degree in Engineering Science and Mechanics from the Pennsylvania State University. After graduating from college, I attended Duke University in Durham, NC, as a graduate student of Prof. Nan M. Jokerst in the Electrical and Computer Engineering department. In 2008, I received a master of science degree in Electrical Engineering from Duke University.
Publications


Honors