Chip Scale Integrated Optical Sensing Systems

with Digital Microfluidic Systems

by

Lin Luan

Department of Electrical and Computer Engineering
Duke University

Date:_______________________

Approved:

___________________________
Nan M. Jokerst, Supervisor

___________________________
April S. Brown

___________________________
Richard B Fair

___________________________
Anne Lazarides

___________________________
Tomoyuki Yoshie

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

2010
ABSTRACT

Chip Scale Integrated Optical Sensing Systems

with Digital Microfluidic Systems

by

Lin Luan

Department of Electrical and Computer Engineering
Duke University

Date: ____________________

Approved:

_________________________
Nan M. Jokerst, Supervisor

_________________________
April S. Brown

_________________________
Richard B Fair

_________________________
Anne Lazarides

_________________________
Tomoyuki Yoshie

An abstract of a dissertation submitted in partial fulfillment of the requirements for the degree of Doctoral of Philosophy in the Department of Electrical and Computer Engineering in the Graduate School of Duke University

2010
Abstract

Data acquisition and diagnostics for chemical and biological analytes are critical to medicine, security, and the environment. Miniaturized and portable sensing systems are especially important for medical and environmental diagnostics and monitoring applications. Chip scale integrated planar photonic sensing systems that can combine optical, electrical and fluidic functions are especially attractive to address sensing applications, because of their high sensitivity, compactness, high surface specificity after surface customization, and easy patterning for reagents. The purpose of this dissertation research is to make progress toward a chip scale integrated sensing system that realizes a high functionality optical system integration with a digital microfluidics platform for medical diagnostics and environmental monitoring.

This thesis describes the details of the design, fabrication, experimental measurement, and theoretical modeling of chip scale optical sensing systems integrated with electrowetting-on-dielectric digital microfluidic systems. Heterogeneous integration, a technology that integrates multiple optical thin film semiconductor devices onto arbitrary host substrates, has been utilized for this thesis. Three different integrated sensing systems were explored and realized. First, an integrated optical sensor based upon the heterogeneous integration of an InGaAs thin film photodetector with a digital microfluidic system was demonstrated. This integrated sensing system
detected the chemiluminescent signals generated by a pyrogallol droplet solution mixed with H₂O₂ delivered by the digital microfluidic system.

Second, polymer microresonator sensors were explored. Polymer microresonators are useful components for chip scale integrated sensing because they can be integrated in a planar format using standard semiconductor manufacturing technologies. Therefore, as a second step, chip scale optical microdisk/ring sensors integrated with digital microfluidic systems were fabricated and measured. The response of the microdisk and microring sensing systems to the change in index of refraction, due to the glucose solutions in different concentrations presented by the digital microfluidic to the resonator surface, were measured to be 95 nm/RIU and 87 nm/RIU, respectively. This is a first step toward chip-scale, low power, fully portable integrated sensing systems.

Third, a chip scale sensing system, which is composed of a planar integrated optical microdisk resonator and a thin film InGaAs photodetector, integrated with a digital microfluidic system, was fabricated and experimentally characterized. The measured sensitivity of this sensing system was 69 nm/RIU. Estimates of the resonant spectrum for the fabricated systems show good agreement with the theoretical calculations. These three systems yielded results that have led to a better understanding of the design and operation of chip scale optical sensing systems integrated with microfluidics.
This dissertation is dedicated

to my mother Yuying Shi and my father Lumin Luan for their endless love and support

through the years and to my husband Guanxiong Xu, who is with me
# Contents

Abstract ........................................................................................................................................ iv

List of Tables .................................................................................................................................. x

List of Figures .................................................................................................................................. xi

Acknowledgements ............................................................................................................................ xvi

Chapter 1 Introduction .................................................................................................................... 1

1.1 Statement of Problem/Motivation ............................................................................................ 1

1.2 Approach .................................................................................................................................. 2

Chapter 2 Background .................................................................................................................... 5

2.1 Optical Microresonators ........................................................................................................ 5

2.1.1 Introduction of Optical Microdisk/ring Resonators ............................................................ 5

2.1.2 Review of Microresonator Technologies ........................................................................... 6

2.1.3 Optical Microresonator Sensors ....................................................................................... 8

2.2 Digital Microfluidic Systems ................................................................................................ 10

2.3 Heterogeneous Integration .................................................................................................... 14

2.3.1 Heterogeneous Integration of Thin Film Metal-Semiconductor-Metal Photodetectors Onto Host Substrate ............................................................................................. 15

2.3.2 Chip Scale Heterogeneous Integration of Thin Film MSM PD with Planar Photonic Structures ..................................................................................................................... 16

Chapter 3 Design of Components and Systems for Photonic and Microfluidic Integration .......... 17

3.1 Introduction to the Optical Microring/disk Resonator Sensors .............................................. 17

3.1.1 Microresonator Sensing Schemes ....................................................................................... 17
3.1.2 Sensing Mechanisms........................................................................................................ 18
3.1.3 Microresonator Calculations Based on Waveguide Theory ......................... 20
3.1.4 Calculations Based on Conformal Transformation Method ......................... 26
3.1.5 Finite-Difference Time-Domain (FDTD) Method ........................................ 31
3.1.6 Detection Limit (DL) and Quality (Q) Factor ............................................... 32
3.1.7 Free Spectral Range (FSR) ........................................................................ 34
3.2 Design of Digital Microfluidic Systems............................................................... 35
3.3 Design of Integrated Optical Sensing Systems................................................... 36
  3.3.1 Design of an Integrated Chemiluminescence Sensor in a Digital Microfluidics Platform ..................................................................................................................... 36
  3.3.2 Design of a Chip Scale Integrated Optical Microring/disk Sensor with a Digital Microfluidics System ...................................................................................................... 38
  3.3.3 Design of an Integrated Optical Microresonator Sensor with a Thin-film Semiconductor Photodetector and EWD Microfluidic system .................................. 40
Chapter 4 Fabrication and Test ................................................................................. 44
  4.1 Fabrication and Test of Microresonators ............................................................ 44
    4.1.1 Configurations of Microdisk Resonators ...................................................... 44
    4.1.2 Fabrication of Microdisk Resonators ......................................................... 46
    4.1.3 Measurement Results ............................................................................. 51
  4.2 Fabrication and Test of Digital Microfluidic systems ....................................... 55
  4.3 Fabrication and Test of Thin Film MSM Photodetectors .................................. 57
  4.4 Fabrication and Test of Integrated Chip Scale Sensing Systems .................... 60
    4.4.1 Integrated Chemiluminescence Sensor in a Digital Microfluidic Platform ² 60
4.4.1.1 Fabrication and System Integration ................................................................. 60
4.4.1.2 Measurement Results 2 ................................................................................. 62
4.4.2 Optical Microresonator Sensors Integrated with EWD Digital Microfluidic Systems .................................................................................................................. 67
  4.4.2.1 Fabrication and System Integration .............................................................. 67
  4.4.2.2 Experimental Measurements ..................................................................... 71
4.4.3 Chip Scale Integrated Optical Microresonator Sensors with Embedded Thin-Film Photodetector on EWD Digital Microfluidics Platforms ........................................... 83
  4.4.3.1 Fabrications and System Integration .............................................................. 83
  4.4.3.2 Experimental Measurements ..................................................................... 88
  4.4.3.3 Measurement Results and Data Analysis ...................................................... 90
Chapter 5 Experiment vs. Theory ............................................................................. 95
  5.1 Characterization of Microresonators and Comparison to Experiments .......... 95
  5.2 Sensitivity of Microresonator Sensors and Comparison to Experiments ....... 101
  5.3 Coupling Efficiency from Waveguide to PD and Comparison to Experiments.. 105
Chapter 6 Conclusions and Future Work ................................................................. 110
  6.1 Conclusions ....................................................................................................... 110
  6.2 Future Work ...................................................................................................... 113
References ................................................................................................................ 115
Biography .................................................................................................................. 122
List of Tables

Table 3.1: Thickness vs. spin speed data for selected SU-8 2000 resists. (Referred from NANO™ SU-8 2000 Negative Tone Photoresist Formulations 2002-2035 of MICRI CHEM) ................................................................. 25

Table 3.2: Refractive index of the materials used for waveguide and photodetector ...... 43

Table 4.1: Material structures used for large-area high-speed I-MSMs grown by Dr Brown’s group ........................................................................................................... 58

Table 5.1: The resonant peaks recorded from the output spectrum shown in Fig. 4.16 (DI water) and the comparisons to the resonant wavelength (between 1560-1565 nm) calculated by the conformal transformation method. ................................................................. 97

Table 5.2: The resonant peaks recorded from the output spectrum shown in Fig. 4.19 (DI water) and the comparisons to the resonant wavelength calculated by the conformal transformation method. .................................................................................................................. 99

Table 5.3: The calculated coupling efficiency from the waveguide to the PD as the variation of the coupling loss the waveguide to the pigtailed fiber. ................................. 109
List of Figures

Figure 2.1 Schematic of a planar microdisk resonator showing an input broad linewidth optical signal, and the resultant output signals. ................................................................. 6

Figure 2.2 Side view of a co-planar electrowetting system ........................................... 12

Figure 3.1 Two sensing mechanisms: homogeneous sensing (left) and surface sensing (right). ................................................................. 20

Figure 3.2 Overall sensitivity plotted as a function of W and Rc for (a) TE and (b) TM fundamental modes in the case of homogeneous sensing. .............................................. 24

Figure 3.3 Configuration of a microdisk cavity. ................................................................. 28

Figure 3.4 Overall sensitivity plotted as a function of W and $R_c$ for TE fundamental modes based on conformal transformation method ...................................................... 31

Figure 3.5 Side view of a co-planar electrowetting chip integrated with a thin film photodetector. The top plate is customized and integrated with a photodetector by the author. ................................................................. 38

Figure 3.6 Scheme of an optical microresonator sensor integrated with a digital microfluidic system. ................................................................. 39

Figure 3.7 (a) Side view of an integrated chemical optical microdisk sensor integrated with an electrowetting chip and photodetector; (b) Integrated microdisk sensor with thin film InGaAs MSM photodetector. ................................................................. 42

Figure 4.1 Schematic cross sections of the two types of microdisk resonators demonstrated within this thesis, and photomicrographs of each resonator (a) A schematic cross section and photomicrograph of a microdisk resonator with the input/output waveguide integrated on top of the microdisk structure; (b) A schematic cross section and photomicrograph of a microdisk resonator with the input/output waveguide integrated partially under the microdisk structure. 63 ...................................................... 45

Figure 4.2(a) Scanning electron micrograph image of microdisk curved edge (right) which is buried underneath the straight waveguide (left). 63 ...................................................... 47
Figure 4.2(b) Scanning electron micrograph image of the microdisk curved edge (left) with straight waveguide (right) buried underneath the microdisk........................................48

Figure 4.3 Vertically coupled microdisk resonator with red light optical excitation at a wavelength of 660 nm. 63 ............................................................................................................................... 51

Figure 4.4 Schematic view of the experimental setup for measuring the output spectrum of microdisk resonators. 63 ............................................................................................................................... 52

Figure 4.5(a) Measured output spectrum of a microdisk structure with the microdisk on the bottom and the waveguide on top, corresponding to the structure in Fig. 4.1a; (b) Measured output spectrum of a microdisk structure with the microdisk on top with a buried waveguide, corresponding to the structure in Fig. 4.1b. 63 ........................................................................................ 54

Figure 4.6 Cross section and a photomicrograph of an EWD based digital microfluidic system. ............................................................................................................................... 56

Figure 4.7 Series of photographs showing how the dyed water droplets were actuated and dispensed from the reservoir, and were moved and mixed along the channel. ....... 57

Figure 4.8 (a) Top view of MSM photodetector metal fingers deposited on the InGaAs substrate (b) Top view of MSM photodetector metal fingers after mesa etching; (c) MSM thin film devices after substrate removal (viewed from the backside): these thin film devices are 1 micron thick, and the metallized finger/pad surface shown in Figure 1 is on the other side of the devices; (d) Thin film MSM photodetector on a transfer diaphragm. 2 ............................................................................................................................... 60

Figure 4.9 Top views of the electrowetting system with a integrated thin film InGaAs MSM photodetector, showing filled reservoirs and a linear array of electrodes. The photodetector was aligned over the array near the reservoir with the H2O2 solution. 2 ... 62

Figure 4.10 Comparisons of measured photocurrent by photodetector when droplet mixed with/without microfluidic system. 2 .......................................................................................... 64

Figure 4.11 Chemiluminescent optical signal sensed by the thin film photodetector integrated with the electrowetting system. Two droplets were first dispensed from their reservoirs, mixed together, then moved under the photodetector. The chemiluminescent droplet was next moved away from the photodetector, moved back under the photodetector again, and then, finally, moved away from the photodetector. The
photodetector output current reflects this optically chemiluminescent droplet movement.  

Figure 4.12 The optical microresonator sensor integrated with a digital electrowetting microfluidic system: (a) photomicrograph of the 600 μm diameter microring resonator with the input and drop (output) waveguides; (b) photograph of the optical microring sensor bonded to a glass carrier and pigtailed (external optical input/output fibers attached to the integrated sensor for testing); (c) photograph of the microfluidic system with which the microresonator sensor was integrated (photo before integration); (d) photomicrograph of the microfluidic system addressing the top plate via with a water/red dye droplet; the droplet was actuated from the reservoir to the via position, and subsequently extruded from the via; (e) photograph of the optical microring resonator sensor (shown in Figure 4.12a) integrated with the digital electrowetting microfluidic system (shown in Figure 4.12c). ........................................................................................................70

Figure 4.13 Setup used for fiber pigtailling the optical fiber to the microresonator sensors. ................................................................................................................................................71

Figure 4.14(a) Spectral measurement setup for the integrated microdisk sensor/microfluidic system: an optical broadband amplified spontaneous emission source and an optical spectrum analyzer. ..................................................................................72

Figure 4.14(b) Spectral measurement setup for the integrated microring sensor/microfluidic system: a tunable diode laser and a photodetector.................................73

Figure 4.15(a) Measured spectral shift of the vertically coupled microdisk resonator sensor as a function of index of refraction (through glucose concentration) presented to the sensor surface. Solid line (Pt. A) de-ionized water with refractive index ~1.3330; long-dashed line (B) 1.36 g/100ml glucose solution with refractive index at ~1.3349; short-dashed line (C) 3.46 g/100ml glucose solution with refractive index ~1.3378; long/short dashed line (D) 4.29 g/100ml glucose solution with refractive index ~1.339..................76

Figure 4.15(b) Resonant wavelength shifts in the microdisk sensor for different concentrations of D-glucose in DI water. ..................................................................................77

Figure 4.16(a) Measured spectral shift of the vertically coupled microring resonator sensor as a function of index of refraction (through glucose concentration) presented to the sensor surface. Solid line (Pt. A) de-ionized water with refractive index ~1.3330; long-dashed line (B) 0.2 g/100ml glucose solution with refractive index at ~1.3338; short-
dashed line (C) 1 g/100ml glucose solution with refractive index ~1.3344; long/short dashed line (D) 2.45 g/100ml glucose solution with refractive index ~1.3364.

Figure 4.16(b) Resonant wavelength shifts in the microring sensor for different concentrations of D-glucose in DI water.

Figure 4.17 Photomicrographs (a and b) and schematic (c) of the optical microresonator sensor integrated with a MSM thin film photodetector: (a) photomicrograph of the 600 μm diameter microdisk resonator with the input and drop (output) waveguides; (b) photograph of the MSM thin film photodetector embedded in an optical waveguide; (c) schematic figure which shows the dimensions of the photodetector features.

Figure 4.18 Photographs of the optical microresonator sensor integrated with a thin film InGaAs photodetector; (a) Top view of the chip, with a 600 μm diameter microdisk resonator integrated with a thin film photodetector; (b) Photograph of the optical microring sensor bonded to a glass carrier and pigtailed. The external optical input fiber and output electronic wires are attached to the integrated system for testing.

Figure 4.19 Spectral measurement setup for the integrated microdisk sensor/microfluidic system: a tunable diode laser and a current/voltage measurement part. Blue lines mean optical connections, and black are electrical connections.

Figure 4.20(a) Measured spectra shift of the vertically coupled microdisk resonator sensor as a function of index of refraction (through glucose concentration) presented to the sensor surface. Solid line (Pt. A) de-ionized water; long-dashed line (B) 0.33 g/100ml glucose solution; short-dashed line (C) 1 g/100 ml glucose; long/short dashed line (D) 1.8 g/100 ml glucose solution.

Figure 4.20(b) Resonant wavelength shifts in the microdisk sensor for different concentrations of D-glucose in DI water.

Figure 5.1 Normalized output spectrum of the optical microdisk resonator (the solid red line shows the measured output spectrum, and the dotted line shows the calculated data).

Figure 5.2 Normalized output spectrum of the optical microdisk resonator (the solid red line shows the measured output spectrum, and the dotted blue line shows the calculated data).
Figure 5.3 Comparisons of the calculated and measured sensitivities plotted as a function of the height of the waveguide for TM0 modes (the blue stars represent the measured sensitivities of the microdisk and microring sensors, and the solid line shows the calculated). ................................................................. 103

Figure 5.4 Comparisons of the measured and calculated normalized output spectrum shifts of a chip scale integrated optical microdisk/PD sensor with 600 μm diameter: The solid line shows the measured spectrum shifts in the experiments from 0wt% water (blue line) to 1wt% (red line) glucose solution. And the dotted lines show the calculated spectrum. ................................................................. 105

Figure 5.5 Simulation results for the coupling loss from the waveguide to the photodetector with FDTD: the optical power monitored before and after the PD are shown on the left; the 3D structure lay out of the waveguide and PD is shown on the right. ................................................................. 107
Acknowledgements

I would like to thank my thesis advisor, Dr. Nan Marie Jokerst for her tremendous support, patience, understanding and help throughout my graduate study experience. She made this research opportunity available to me, for providing valuable insight and guidance on my research, giving me immeasurable hours spent reviewing my technical papers. I would also like to acknowledge Dr. April Brown, Dr. Richard B Fair, Dr. Anne Lazarides and Dr. Tomoyuki Yoshie for serving on my Ph.D. examination committee.

I would like to thank Matthew Royal, Randal Evans, and Sabarni Palit for their helps in my PhD research.

I must acknowledge my group members and my friends for all of the good times and good discussions. Finally I would like to thank the SMIF cleanroom staff for their help and support.
Chapter 1 Introduction

1.1 Statement of Problem/Motivation

Low cost, rapid, miniaturized and portable sensing of chemical and biological analytes is critical to applications in the areas of medicine, environmental monitoring, and security. Chip scale integrated photonic sensing systems which are light, small, low power, and sensitive can be realized with the heterogeneous integration of thin film optical components and with microresonators. The integration of planar photonics sensing systems with digital microfluidics systems is attractive, since the digital microfluidics systems can programmably control the movement of liquid samples and present them to the integrated optical sensing system. The technology of heterogeneous integration provides the possibility of the integration of optical sensing systems with digital microfluidic systems. ¹

Planar optical sensors, especially optical evanescent-wave (EWS) sensors, detect refractive index changes in the sensing area probed by the evanescent waves, which cause corresponding output resonant spectral shifts or power changes. Because of their high sensitivity, robustness, compactness and compatibility, high surface specificity after surface customization, and easy patterning for reagents, EWS optical sensors are becoming increasingly attractive for microfluidic sensing applications. They can also be integrated with electronic and photonic devices, especially with microfluidic systems for sample handling and preparation.

Digital microfluidic lab-on-a-chip (LoC) technology offers droplet-based control of small volumes of fluid (e.g. in micron or pico liter scale) for diagnostic sample
preparation processes, which include droplet dispensing, actuation, and mixing. Currently, much of the published work on electrowetting-based LoC microfluidic devices has been focusing on miniaturization of analytical methods and protocols for the purpose of improving performance and throughput. ²

Because of the difficulty to integrate complex optical functions into microfluidic systems, most digital microfluidic platforms incorporate limited optical capabilities for integrated sensing. However, to date only a few results have been reported on integrating the backend function of optical detection. Part of the reason is the requirement that detector integration must not interfere with electrowetting-based droplet transport, and part is that the heterogeneous integration of thin film (microns thick) photonic components is an emerging technology that is just beginning to be applied to digital microfluidic systems. ²

1.2 Approach

Integrated optoelectronics, with the goal to constitute miniaturized and compact optical devices with high functionality on a single substrate, ³,⁴ is presently attracting a great deal of interest in the application area of integrated chip-scale Bio/Chemical sensing systems. Heterogeneous integration is a crucial technology to integrate thin film optical devices with electronic and fluidic functions to realize chip-scale integrated optical sensing systems. The integration of optical sensors with digital microfluidic systems at the chip scale has the potential to realize portable, miniature, high sensitivity, and self-contained sensing systems for on-chip sample processing. The intimate integration of sensors and digital microfluidics systems are applicable for sample preparation and
diagnostics for biological and chemical sensing in medical diagnostic and environmental monitoring.2

This thesis describes the design, simulation, fabrication, and measurement of the integrated chip scale optical microresonator sensing systems, and also explores the integration of optical microresonator sensors with digital microfluidic systems. This thesis demonstrates the chip scale integration of optical microresonator sensors, waveguides and thin film metal-semiconductor-metal (MSM) photodetectors with electrowetting-on-dielectric (EWD) digital microfluidic systems. First, an integrated optical sensor based upon the heterogeneous integration of an InGaAs thin film photodetector with a digital microfluidic system has been completed for the research in this thesis, and is presented herein. This demonstration of the integration and operation of an active optical device with a microfluidic system is the first step toward the integration of entire optical sensing systems with microfluidic systems. Next, chip scale optical microdisk/ring sensors integrated with digital microfluidic systems were demonstrated and tested. Finally, a chip scale integrated sensing system, which is composed of a planar optical sensing system and a digital microfluidic system was realized.

Chapter 2 provides the background information about optical microresonator sensors and EWD based digital microfluidics. Chapter 3 covers the design of the chip scale integrated planar optical sensing systems and the digital microfluidic systems. In Chapter 4 fabrication processes and the corresponding measurement results from the chip scale integrated sensing systems are detailed. Chapter 5 compares some of the
experimental results to theory. The final chapter summarizes the goals of this research and contributions made to the field. It also points out how this research can be extended in the future.
Chapter 2 Background

2.1 Optical Microresonators

Optical microresonators, such as microdisk or microring resonators, are useful as fundamental blocks in optical systems,\textsuperscript{5} such as lasers\textsuperscript{6-8}, filters\textsuperscript{9-11}, and sensors\textsuperscript{12-15}. For biological and chemical sensing, optical microresonator sensors are attractive because of their high sensitivity, and also the large net dynamic range in arrays for quantitative sensing. In particular, the good detection limit of optical microresonator sensors is attractive due to their high quality factor (Q),\textsuperscript{16} which can lead to very high sensitivity. Moreover, since microresonator sensors have a long optical interaction length from confinement of light in the microcavity at resonance, high sensitivity can be realized in a micron-scale sized device. For example, in contrast to typical interferometric waveguide sensors, microdisk resonators are predicted to improve sensitivity and reduce the amount of analyte needed for detection by at least one order of magnitude.\textsuperscript{17}

2.1.1 Introduction of Optical Microdisk/ring Resonators

Microring/disk resonators consist of three parts: the input and output waveguides (throughput waveguide and drop waveguide), and a microdisk/ring cavity, as shown in Fig.2.1. In this figure, two orthogonal straight optical waveguides serve respectively as the optical input/throughput and as the optical drop output for the resonator. Wavelengths from a broadband optical signal (in the wavelength spectrum) from the input optical waveguide that are on resonance with the microresonator disk will be coupled into the microdisk cavity through evanescent wave coupling, exciting whispering-gallery modes\textsuperscript{18, 19} in the disk cavity, which are then coupled into the drop waveguide by evanescent
waves. The off-resonance frequencies are transmitted in the throughput waveguide, and appear at the port of the throughput waveguide. In a microcavity resonator, the guided waves meet the resonance condition when their round-trip phase is equal to an integer multiple of $2\pi$.

Figure 2.1 Schematic of a planar microdisk resonator showing an input broad linewidth optical signal, and the resultant output signals.

### 2.1.2 Review of Microresonator Technologies

Materials used to fabricate microresonators include various glasses and polymers (on a silica-on-silicon substrate), silicon-on-insulator, gallium arsenide, and indium phosphide. Polymer microresonators are attracting a great deal of interest, because polymer materials are inexpensive and have relatively simply fabrication and planarization processes, especially when photosensitive polymers are used. In addition, polymers typically have good optical transparency (i.e., relatively low optical loss, although glass and semiconductors can be better) and relatively low refractive index.
(typically in the range of 1.44-1.65). Polymers also offer such advantages as low scattering loss due to the surface roughness because of the low refractive index. Polymer microresonators can be integrated with other passive planar communication components and with active thin film optoelectronic components for applications such as fully integrated chip-scale optical sensing. As reported, polymer microcavity devices have been integrated with InGaAs photodetectors on silicon (Si). 20, 21

Two major coupling geometries have been reported to date for the optical power coupling from the waveguide to the microcavity: lateral coupling and vertical coupling. For the laterally coupled microcavity structure, the coupling waveguides and the microcavity lie in the same plane, and the optical signal is coupled through the gap between the waveguide and microcavity by evanescent waves. Electron beam lithography (EBL) 22 and nanoimprint lithography (NIL) 23 are two major technologies used to fabricate laterally coupled microresonators. Electron beam lithography is a typical method of patterning structures in the nanometer scale dimension. Most laterally coupled microdisk or microring resonators are fabricated by electron beam lithography. NIL is a more high-throughput method of creating substrates with nanoscale features. NIL consists of two steps: imprint and pattern transfer. For example, Chao, C.-Y. et al. used a combination of e-beam lithography, nanoimprint lithography, and reactive ion etching (RIE) to fabricate a hard mold to directly imprint a polymer film to form optical waveguides in microring devices. 24 However, the two methods described above do not lead to low-cost mass manufacture of multiple sensors in large arrays. Therefore, it is cost and throughput prohibitive to fabricate arrays of microresonators in low cost polymers for
chip-scale sensing using EBL or NIL. An attractive alternative in a vertically coupled microcavity structure, where the coupling waveguides and the microcavity do not lie in the same plane, rather, one on top of the other. The optical signal is coupled through the middle cladding material between the waveguide and resonator. Herein, how to fabricate vertically coupled microresonators with simple and inexpensive photolithographic techniques will be described later in Chapter 4.

2.1.3 Optical Microresonator Sensors

Planar microresonators are attractive for chip scale integration with planar photonic components in chip scale integrated sensing systems. Because they are compact in size, simple to fabricate, and surface selectable for targeted sensing, planar microresonators enables large sensing arrays using standard micro/nano fabrication methods, as well.

Polymer microresonators can be integrated with other passive and active planar optical components for applications, such as fully integrated chip scale optical sensing. For instance, polymer microcavity devices have been integrated with InGaAs photodetectors on silicon.\textsuperscript{25, 26} In addition, the planar and compact structure of microresonators makes it possible to integrate with other electrical or mechanical systems (such as planar optical circuits, Si CMOS circuits or a microfluidic system). Planar microresonator sensors have already been reported to be used for chemical sensing applications, such as glucose, oxylene, and toluene in water.\textsuperscript{13, 21, 27, 28} Moreover, optical microresonator sensors, which consist of vertically coupled polymer microring resonator sensors and a chemically selective polymer membrane surface coating, have also been
demonstrated selectively chemical sensing by surface customization. The surface functionalization, which provided selective permeability for target chemicals, enables chemical analyte discrimination on the microresonator sensors surface. Likewise, optical microresonators can be used for biosensing, which includes clinical screening, medical diagnostics, and screening of chemical compounds. Optical microresonators enable strong interactions between optical light and the bonded analyte, resulting in ultrasensitive optical detection. Therefore, label-free biosensing with optical microresonators has been considered to be very promising, not only because of their high sensitivity, but also for the potential for integration in multidimensional arrays. Optical microcavities have been recently reported for single biomolecule and virus detection, using a highly confined microscale mode volume and ultrahigh quality factor.

In conclusion, polymer microresonators are attractive components used for miniaturized, portable chip scale integrated sensing systems for medical and environmental diagnostics and monitoring. Vertically coupled microresonators, as described before, can be simply fabricated by standard photolithography, which enables mass fabrication for low cost sensing systems. Microresonators can also be integrated with planar optical system components, such as polymer waveguides and thin film photodetectors, onto host substrate using heterogeneous integration, to realize planar chip scale integrated sensing systems.
2.2 Digital Microfluidic Systems

Lab-on-a-chip (LoC) technology enables integration of multiple functional devices at the millimeter or smaller scale on a single chip to realize multiple fluidic processes, including manipulating and mixing droplets of liquid in volumes of microliter or less. It provides the possibility of functional miniaturization at the micro-scale for multiple areas applications, including: chemistry, biology, bioengineering, physics, electronics, clinical/medical science, chemical engineering, and materials science. Advantages include compactness, increased automatization, power economization, and high throughput for mass production. So lab-on-a-chip technology can be widely applied for chemical sensing and biological diagnostics. Lab-on-chip technology can lead to portable, compact, easy to integration with optical devices, and realization of complicated sample processing.²

Microfluidic lab-on-a-chip technology, which allows miniaturization and integration of complex lab functions for sample preparation and detection, offers one possible solution to move sophisticated diagnostic tools out of large laboratories. They are inexpensive, but also accurate, reliable, and well suited for medical applications in the developing world. Microfluidics-based devices can perform tests at sensitivity, specificity and reproducibility levels similar to those of large laboratory equipment, but only require sample. However, the cost of a hypothetical microfluidics-based diagnostic tool must be extremely low if we want it to be widely applicable. There are many potential benefits of lab-on-chip diagnostic devices and systems for specific applications, which include: access to diagnostic tools not previously available, and thus conduct faster/more accurate
diagnoses; better utilization of minimally trained healthcare workers; and better use of existing therapeutics. 34

There are several methods for independently manipulating digital microfluidic droplets, including electrowetting 35, dielectrophoresis 36, and thermocapillarity 37. Electrowetting microfluidics has become attractive in many applications because it is fast, has low power consumption, and is scalable. 38 In addition, these systems have good volume control, so that droplets can be mixed and split with good concentration control. 39 A wide variety of aqueous solutions can be used, including unprocessed human fluids.40

Electrowetting-on-dielectroic (EWD) microfluidic systems enable electrically actuation of droplets of up to several microliters by modulating the interfacial tension between a liquid and an electrode coated with a dielectric layer. EWD is a phenomenon that demonstrates the modification of wetting behavior for a polarizable and/or conductive liquid droplet by an electrical field when the liquid is in contact with hydrophobic, insulated electrodes. The electric field established between the dielectric layer and the ground results in an imbalance of the interfacial tension on the droplet surface if the electric field is applied to only one portion of the droplet, which forces the droplet to move. 2 The droplet is sandwiched between two hydrophobic plates, with the top plate grounded, and the bottom plate containing patterned electrodes buried underneath a dielectric, while surrounded by an immiscible fluid (typically oil) to both prevent droplet evaporation and to reduce the electrowetting threshold voltage, as shown
in Figure 2.2. These systems can dispense droplets from reservoirs, mixed or split them rapidly, and transport the droplets quickly.

Figure 2.2 Side view of a co-planar electrowetting system.

For sensing applications, EWD microfluidic systems are advantageous in comparison to typical continuous fluidic system: First, there are no moving parts, such as pumps or valves, since all the operations are carried out under direct voltage control. Second, EWD enables multiple droplets to be controlled independently and simultaneously, since the electrowetting force is localized at the contact surface. Third, in sensing applications, it is possible to realize near 100% utilization of the sample or reagent, since no fluid is wasted for priming channels or filling reservoirs, in contrast to continuous flow microfluidics. And, because it enables the use of glass substrates and some transparent electrodes, such as indium-tin-oxide (ITO), EWD microfluidic systems are compatible for observation with a microscope. Finally, EWD systems are extremely energy efficient: they generally consume nanowatts–microwatts of power per transfer,
since there is almost no electrical current passing through the electrodes. However, there are still some concerns for the realization of chip-scale EWD microfluidic systems. First, a top plate is necessary to dispense liquid droplets, and it also has to be grounded. The co-planar structure, which includes the ground on the bottom plate, can help solve this problem. However, top plates are still needed for droplet dispensing. High dispense voltages (larger than 10 volts), are required to move the droplets. But thinner dielectric layers and new isolation materials will help to decrease the drive voltage. \(^2\)

As mentioned before, this droplet-based EWD mechanism can be equivalently mapped to bench-scale wet chemistry/biology in functionality. Therefore, many of the established assays and protocols in chemistry or biology can be scaled down, automated, integrated and executed on the compact digital EWD microfluidic platforms. And each execution step can be implemented by direct computer control, which allows maximum operational flexibility and minimizes direct human intervention. \(^2\)

However, a great deal of work on EWD digital microfluidics has been focused on miniaturization of analytical methods for the purpose of improving performance and throughput. More research is currently focusing on integrating the backend function of optical detection with the digital microfluidic systems. An integrated optical sensor based upon the heterogeneous integration of an InGaAs-based thin-film photodetector with a digital microfluidic system was demonstrated in 2008, and was the first step toward the heterogeneous integration of an entire planar optical sensing system on an EWD platform.\(^2\)
2.3 Heterogeneous Integration

Nowadays, integrated optics (IO) and optoelectronic devices are increasingly being used for chemical and biological sensing applications. This is mainly due to the advantages that optical devices have in comparison to electrical methods, such as high sensitivity, immunity to electromagnetic interference. One technology in use for optoelectronic integration is heterogeneous integration. Heterogeneous integration is a technology through which optical thin film semiconductor devices (e.g. lasers, light emitting diodes, photodetectors) can be integrated onto arbitrary host substrates including Si$^{41}$, Si CMOS$^{42}$, and FR4$^{43}$. This process selectively removes the growth substrate from the epitaxial layers that constitute the functional device, and the resulting thin film devices can then be transferred and bonded to other host substrates. It can be used to realize optical and electrical function on one chip, which is also known as Planar Lightguide Circuit (PLC) or Silicon Motherboards$^{44}$. Technologies for heterogeneous integration enable the integration of thin film semiconductor devices onto arbitrary host substrates (Si, GaAs, CMOS circuits), and therefore, more complex optical functions can be integrated into other mechanical, biological, and microfluidic systems. As reported herein, heterogeneous integration has been used to integrate optical components with microcavity resonators onto host substrates such as electrowetting microfluidics systems.$^{2}$ The integration of optical sensors with EWD microfluidic systems at the chip scale has the potential to realize portable, miniature, high sensitivity, and self-contained sensing systems that include on-chip sample processing.
2.3.1 Heterogeneous Integration of Thin Film Metal-Semiconductor-Metal Photodetectors Onto Host Substrate

InGaAs based thin film photodetectors can be heterogeneously integrated with other optical components or systems. Thin film MSMs can be conventional or in inverted format (if they are inverted, then they are inverted metal-semiconductor-metal photodetectors (I-MSM PDs)). Conventional vertically illuminated photodetectors are divided into two broad categories: p-i-n/avalanche photodetectors and metal-semiconductor-metal (MSM) photodetectors. MSM photodetectors, with interdigitated Schottky finger contacts, are attractive because they have a lower capacitance per unit area than p-i-n detectors. Thus, MSMs are larger than p-i-n devices operating at the same speed, reducing the cost associated with alignment and packaging. Inverted MSMs (I-MSM) are thin film MSMs with fingers on the bottom of the device, thus eliminating the finger shadowing that causes conventional MSMs to have low responsivity. I-MSMs have been demonstrated that have responsivities that are comparable to p-i-n devices for the same operational speeds. These I-MSMs are typically about 1 micron thick, and have been heterogeneously bonded directly onto Si CMOS circuits, such as a chip-based microprocessor and an analog receiver designed for heterogeneous photodetector integration, thus demonstrating optical interconnection to the microprocessor integrated onto a single Si CMOS integrated circuit. The thin film heterogeneous integration process of MSM PD will be detailed in Chapter 4.
2.3.2 Chip Scale Heterogeneous Integration of Thin Film MSM PD with Planar Photonic Structures

Thin film photodetectors have been integrated with waveguide and coupler structures, on a Si host substrate and interconnected with a polymer waveguide. Embedded thin film PDs in polymer waveguides offer planar high speed interconnections with low power consumption, reliable performance, and high integration density. The optical signal coupling can be either directly or evanescently coupled from the waveguide. The reported optical PD with waveguide use benzocyclobutene (BCB) polymer optical waveguides with embedded InGaAs-based thin film inverted MSM photodetectors, all integrated onto a Si substrate. In addition, a 1x4 thin film MSM photodetector array embedded in a photoimageable polymer multi-mode interference (MMI) coupler has also been demonstrated.

The chip scale integration of thin film photodetectors with waveguides can extend to the integration of these devices with planar optical sensors, as well. A polymer microresonator sensor integrated with a thin film InGaAs photodetector, has been reported. This thin film photodetector with final thickness about 1.26 μm, was embedded in the output waveguide of a vertically coupled microresonator to provide optical to electrical conversion for the output. This photodetector monitors the waveguide output power changes, when sensing occurs on the top of the microresonator sensor.
Chapter 3 Design of Components and Systems for Photonic and Microfluidic Integration

3.1 Introduction to the Optical Microring/disk Resonator Sensors

Microresonator sensors have already been widely explored for biochemical sensing applications. There are several important parameters, including sensitivity and detection limit, which need to be considered in order to design and optimize microresonator sensors. In addition, free spectral range (FSR), quality factor (Q factor) and the signal-to-noise ratio (SNR) are also fundamental and important parameters for microresonator sensors. In the system design, the criteria for an optimized sensing system are how to enhance the sensitivity and improve the detection limit. All parameters above will be discussed herein and the device geometry will be optimized to obtain the desired system performance. We focus on polymer microring resonators sensors in the following design cases.

3.1.1 Microresonator Sensing Schemes

Microresonator sensors can use two different sensing schemes. The first resonant-wavelength-shift scheme monitors the shift in the resonant wavelength caused by detection of the target analytes, which results in a sensitive, high dynamic range sensor. By monitoring multiple resonant points, it also enables the use of statistical methods to reduce noise and to increase signal to noise ratio (SNR). However, this scheme typically requires sophisticated spectral measurement equipment, such as an input tunable laser with a photodetector monitoring the output, or a broadband optical source input with an optical spectrum analyzer to monitor the output. The second intensity-variation
scheme monitors the output optical power changes at a particular wavelength that is located on a highly sloped point in the optical spectrum. Theoretically, this scheme can provide a higher sensitivity and requires a simpler setup (e.g. an optical power meter or photodetector). However, this technique has a smaller dynamic range than the spectral technique, and requires very high system stability. In addition, the detection accuracy can be affected by transverse mode competition in a multimode microresonator. For the integrated system reported in this thesis, spectral measurements will be chosen for more accurate detection. This sensing scheme will include either a tunable laser with a photodetector, or a broadband optical source with an optical spectrum analyzer to acquire the maximum amount of information available from the integrated sensing systems. The prospects for chip-scale integration for either optical measurement scheme system are excellent: for the spectral approach, a tunable semiconductor laser could be integrated with a photodetector, or a high brightness LED with a planar integrated spectrometer with an array of monitor photodetectors to read the spectrometer output. Likewise, the single wavelength monitoring scheme could be implemented using differential sensing to reduce the system noise and to increase the system stability. Differential sensing can be implemented by subtracting the outputs of two identical microresonators, one of which is exposed to the target analyte, and the other is used to track environmental changes. Thus, either optical measurement system could be realized in a chip-scale integrated format.

### 3.1.2 Sensing Mechanisms

In general, there are two sensing mechanisms for optical-waveguide based sensors, including microresonator sensors. They are homogeneous sensing and surface
sensing, as shown in Fig. 3.1. In homogeneous sensing, the device is typically surrounded by the analyte solution to be detected, and the solution is taken as the top cladding layer of the device. Therefore, the changes of the bulk refractive index of the solution are determined by the concentration changes of the analyte distributed in the solution. In this mechanism, all materials, including the detected analyte in the solution, contribute to the effective-index shift; therefore, there is no selectivity for the analyte to be detected.

The other mechanism is surface sensing, which enables surface customization. In surface sensing, the surface of the optical sensor is customized, for example, with a membrane, or pretreated to have binding materials (such as single strand DNA), which can selectively absorb or bind to the specific analyte. These mechanisms can provide higher sensitivity as well as sensing specificity, because of the larger evanescent wave tail and larger refractive index changes at the sensor surface. In the system tests reported herein, glucose solutions with different concentrations are used as analytes to demonstrate the operation of the integrated system and the sensor sensitivity. Therefore, the homogeneous sensing mechanism will be employed in this thesis.
3.1.3 Microresonator Calculations Based on Waveguide Theory

Waveguide theory has already been used to calculate the sensitivity of the microresonator sensors. The calculations of sensitivities help to design suitable ranges of the sensor characteristics (e.g. microring diameter, waveguide height). This procedure facilitates the design and the optimization of planar microresonator sensors.

The sensitivity of a sensing device is defined as the ratio of the change in the measured optical parameter (i.e., resonant wavelength of the guided modes $\lambda_c$ in the resonant-wavelength-shift scheme and optical power in the drop or through waveguide in the intensity-variation scheme) to the change in the waveguide parameter affected by analytes (cladding index $n_{cl}$ in homogeneous sensing and adsorbed film thickness in surface sensing). In this research, we chose the sensing system employing homogeneous sensing with a resonant-wavelength-shift scheme, so, according to these definitions, the sensitivity of the device is

$$S = \frac{\partial \lambda_c}{\partial n_{cl}} = \frac{\partial \lambda_c}{\partial n_{\text{eff}}} \cdot \frac{\partial n_{\text{eff}}}{\partial n_{cl}}. \quad \text{(Eqn. 3.1)}$$

According to Dr Chao’s paper, in contrast to the device sensitivity, which is defined as the ratio of the change in $\lambda_c$ to the effective-index change, the waveguide
sensitivity is defined as the ratio of the effective index change to the change in the waveguide parameter affected by analytes $S_w = \frac{\partial n_{efl}}{\partial n_{cl}}$. Therefore, $S = \frac{\partial \lambda}{\partial n_{efl}} \cdot S_w$.

Assuming for TE modes, with the propagation in the $z$ direction, $E_z = 0$ and $E_x = 0$ in the transverse plane. Where, $W$ is the height of the waveguide, $k_0$ is the wave number in vacuum, $\gamma_c$ is the attenuation coefficients in the cladding, $k_x$ the propagation constant along the transverse direction, and $n_{co}$ and $n_{cl}$ are the equivalent core and cladding indexes, respectively. Furthermore, $n_{efl}$ is effective index and

$$n_{efl} = \sqrt{n^2_{co}k^2_0 - k^2_x} \over k_0$$

When $\gamma^2_c = (n^2_{co} - n^2_{cl})k^2_0 - k^2_x$, by replacing $k_x$ and $\gamma_c$ with $n_{efl}$ in

$$\tan(\frac{k_xW}{2}) = \frac{\gamma_c}{k_x} \text{ for TE or } \tan(\frac{k_xW}{2}) = \frac{n^2_{co}\gamma_c}{n^2_{cl}k_x} \text{ for TM},$$

the waveguide sensitivity for both TE and TM polarizations, can be derived and respectively listed below.

$$\left(S_w\right)_{h, \text{TE}} = \frac{n_{cl}}{n_{efl}} \times \frac{1 + \frac{2\gamma_c k_x}{n^2_{co}k^2_0} \tan(\frac{k_xW}{2})}{1 + \frac{n^2_{co}\gamma_c}{n^2_{cl}k^2_x} \left[ \tan(\frac{k_xW}{2}) + \frac{k_xW}{2} \sec^2(\frac{k_xW}{2}) \right]} \text{ for TE modes}^{49}$$

(Eqn. 3.2)

And

$$\left(S_w\right)_{h, \text{TM}} = \left[ \frac{n_{efl}}{n_{cl}} \times \left[ 1 + \frac{\gamma_c}{k_x} \tan(\frac{k_xW}{2}) + \frac{\gamma_cW}{2} \sec^2(\frac{k_xW}{2}) \right] \right]^{-1} \text{ for TM modes}^{49}$$

(Eqn. 3.3)
When $\lambda_c = \frac{n_{\text{eff}} \cdot 2\pi R_c}{m}$, the microring resonator resonances, where $m$ is an integer representing the resonance order, and $R_c$ is the radius of the microring resonator. And the waveguide sensitivity is correlated to the sensor device sensitivity by the formula $S = S_w \cdot \frac{2\pi R_c}{m}$.

Let us assume such factors, as $k_0$, $n_{co}$, and $n_{cl}$, are fixed, According to the formula (Equ 3.2 and 3.3), for a microring resonator structure the waveguide width $W$ and the radius of microring $R_c$, are the two most important parameters, which decide the waveguide sensitivity, as shown in Fig 3.2. We will optimize $W$ and $R_c$ to obtain the best sensing performance. Based on Dr. Chao’s theory, a detailed calculation of the sensor sensitivity is presented herein. Fig. 3.2 shows the waveguide sensitivity plotted as a function of $W$ (from 2 µm to 10 µm) and $R_c$ (from 400µm to 800 µm) for (a) TE and (b) TM fundamental modes in the homogeneous sensing, when the optical wavelength is at 1560nm, and the refractive index of the core and cladding layer are 1.56 and 1.49, respectively.
Relationship between sensitivity and $W$, $R_c$ for TE$_{00}$

(a)
Figure 3.2 Overall sensitivity plotted as a function of W and Rc for (a) TE and (b) TM fundamental modes in the case of homogeneous sensing.

The optical mode used in the simulation is the TE$_{00}$ mode. Fig. 3.2 shows that higher waveguide sensitivity requires a smaller waveguide width but has little dependence on the radius of the microcavity.

When deriving the waveguide sensitivity in Chang’s paper, the k vector along the radial direction in the ring structure is neglected. Thus, the actual $n_{\text{eff}}$ in experiments is
smaller than what is calculated above. Therefore, the actual sensitivity of the microring resonator sensors in measurements should be smaller than the simulation results. When considering second or higher order modes, the \( n_{\text{eff}} \) will be smaller than that for the fundamental mode. Therefore, the higher order modes are less sensitive than the fundamental mode.

**Table 3.1: Thickness vs. spin speed data for selected SU-8 2000 resists.**

(Referred from NANO™ SU-8 2000 Negative Tone Photoresist Formulations 2002-2035 of MICRI CHEM).

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Viscosity** (cSt)</th>
<th>Thickness** (( \mu \text{m} ))</th>
<th>Spin Speed (rpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SU-8 2002</td>
<td>7.5</td>
<td>2</td>
<td>3000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>1000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5</td>
<td>3000</td>
</tr>
<tr>
<td>SU-8 2005</td>
<td>45</td>
<td>6</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.5</td>
<td>1000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7</td>
<td>3000</td>
</tr>
<tr>
<td>SU-8 2007</td>
<td>140</td>
<td>8.5</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>12.5</td>
<td>1000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10</td>
<td>3000</td>
</tr>
<tr>
<td>SU-8 2010</td>
<td>380</td>
<td>13</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20</td>
<td>1000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15</td>
<td>3000</td>
</tr>
<tr>
<td>SU-8 2015</td>
<td>1250</td>
<td>21</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>38</td>
<td>1000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>25</td>
<td>3000</td>
</tr>
<tr>
<td>SU-8 2025</td>
<td>4500</td>
<td>41</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>75</td>
<td>1000</td>
</tr>
</tbody>
</table>

** Approximate

From the formula derived above, the microresonator sensitivity mainly depends on the transmitted waveguide height. This parameter can be manipulated to achieve a
higher sensitivity. However, decreasing waveguide height will also increase the fabrication challenge. In this thesis, a polymer material SU8 was used as the core layer of the microresonators, the thickness of which is decided by the fabrication process. Based on the data listed in Table 3.1, SU-8 2002 with around 2 µm height was a reasonable choice to use for fabrication.

3.1.4 Calculations Based on Conformal Transformation Method

The conformal transformation method is an important theoretical approach used for calculating whispering gallery modes in microdisk cavities. This gives us an analytical understanding of the modes supported by the microresonator cavity. In this thesis, these calculations are used to estimate the resonant wavelength and the free spectrum range, and also to predict the microresonator spectral peaks shift as the refractive index above the upper cladding layer changes (i.e., to estimate the spectral shift of the resonance to a change in index of refraction, which is measured in Chapter 4).

Cylindrical coordinates were used herein, as is shown in Fig. 3.3. The solution of the scalar function $\psi$ for the wave equation is:

$$\frac{\partial^2 \psi}{\partial r^2} + \frac{1}{r} \frac{\partial \psi}{\partial r} + \frac{1}{r} \frac{\partial^2 \psi}{\partial \phi^2} + \frac{\partial^2 \psi}{\partial z^2} + k^2 \psi = 0$$  \hspace{1cm} (Eqn. 3.4)

And $\psi = F(r) \exp(\pm im\phi) \exp(ik_z z)$  \hspace{1cm} (Eqn. 3.5)

Where $m$ is an integer of the azimuthal number, $k_z$ is the $z$ direction factor of the $k$ vector, which corresponds to different resonant modes in the $z$ direction and can be referred to as planar waveguide modes $p$ ($p=0,1,2,\ldots$). Note that, $k_z$ is determined by the
given disk thickness, and the structure of the microdisk. Here, \( k_z \) can be solved according to the limit of the boundary conditions in the planar waveguide. \(^{51}\)

Assuming \( \phi(r, \varphi) = F(r) \exp(\pm im\varphi) \), the 2-D scalar wave equation for \( \phi(r, \varphi) \) is

\[
\frac{\partial^2 \phi}{\partial r^2} + \frac{1}{r} \frac{\partial \phi}{\partial r} + \frac{1}{r^2} \frac{\partial^2 \phi}{\partial \varphi^2} + q^2 \phi = 0 \quad (\text{Eqn. 3.6})
\]

Where \( q^2 = (k_1^2 - k_z^2) \) for \( r < R \), and \( q^2 = (k_2^2 - k_z^2) \) for \( r > R \)

Let \( \xi = h_1 W \), and \( k_z^2 = h_i^2 = k_i^2 - \beta^2 \), where, \( \beta \) is the propagation constant, and the eigenvalue equation for the TE mode can be expressed as

\[
\tan \xi = \frac{(V^2 - \xi^2)^{1/2} + \left[ (1 + \alpha_k) V^2 - \xi^2 \right]^{1/2}}{(1 + \alpha_k) V^2 - \xi^2} \quad (\text{Eqn. 3.7})
\]

The eigenvalue equation for the TM mode can be expressed as

\[
\tan \xi = \frac{n_1^2 n_2^2 (V^2 - \xi^2)^{1/2} + n_2^2 \left[ (1 + \alpha_M) V^2 - \xi^2 \right]^{1/2}}{n_2^2 n_3^2 \xi^2 - n_1^2 (V^2 - \xi^2)^{1/2} \left[ (1 + \alpha_M) V^2 - \xi^2 \right]^{1/2}} \quad (\text{Eqn. 3.8})
\]

Where, \( V = \frac{2\pi}{\lambda} W \sqrt{n_1^2 - n_2^2} \), \( \alpha_k = \frac{n_2^2 - n_3^2}{n_2^2 - n_1^2} \), \( \alpha_M = \frac{n_4^2 n_2^2 - n_3^2}{n_3^2 n_2^2 - n_3^2} \), \( n_1 > n_2 > n_3 \) \(^{54}\)
In the vertical z direction, there are only two modes, TE\textsubscript{00} and TM\textsubscript{00}.

After conformal transformation, the wave equation in the (u,v) coordinate system is

\[
\frac{\partial^2 \phi}{\partial u^2} + \frac{\partial^2 \phi}{\partial v^2} + q^2 \exp\left(\frac{2u}{R_c}\right) \phi = 0 \tag{Eqn. 3.9}
\]

where \( u = R \ln\left(\frac{r}{R_c}\right), v = R \varphi \).

\[
k_v^2(u) = (k_1^2 - k_z^2) \exp(2u / R) - k_v^2 , \text{ for } r < R_c \tag{Eqn. 3.10}
\]

And \( k_v^2(u) = (k_2^2 - k_z^2) \exp(2u / R) - k_v^2 \) (Eqn. 3.11) for \( r > R_c \), and \( k_v \) should satisfy the condition for guided mode \((k_2^2 - k_z^2) < k_v^2 < (k_1^2 - k_z^2)\). Let us define the
effective index $n_u$ with $k_u = 2\pi n_u / \lambda$. $k_u$ is determined in the Wentzel-Kramers-Brillouin (WKB) approximation, and, results in the equation:

$$2\sqrt{n_i^2 - n_z^2} \frac{R}{\lambda} (\sqrt{1 - a^2} - a \cos^{-1} a) + \frac{1}{4} \tan^{-1} \sqrt{1 - a^2 / (a^2 + b^2)}, l = 0, 1, 2...$$

(Eqn. 3.12)

Where $a$ and $b$ are defined by $a = n_i / \sqrt{n_i^2 - n_z^2}$, and $b = \sqrt{n_i^2 - n_z^2} / \sqrt{n_i^2 - n_a^2}$, with $n_a = \sqrt{n_i^2 - n_z^2 - n_u^2}$.

The azimuthal mode number $m$ determines the number of modes along the $\phi$ direction. Since, $m = \frac{2\pi n R}{\lambda}$, and $m$ is an integer, for a given microdisk structure, a resonance wavelength corresponds to each $m$.

$$\lambda = \frac{2\pi n R}{m}$$

Since $\lambda$, the sensitivity of the device is

$$S = \frac{\partial \lambda}{\partial n_c} = \frac{2\pi R}{m} \frac{\partial n_c}{\partial n_c}$$

(Eqn. 3.13)

According to the formula derived above, for a microring resonator structure the waveguide width $W$ and the radius of microring $R$, are the two most important parameters, which decide the waveguide sensitivity. We search for the optimal $W$ and $R$ to obtain the best sensing performance. Based on conformal transformation theory, a detailed calculation of the sensor’s sensitivity is presented herein. Figure 3.4 shows the waveguide sensitivity plotted as a function of $W$ (from 2 $\mu$m to 2.2 $\mu$m) and $R$ (from 400$\mu$m to 800 $\mu$m) for TE fundamental modes in the homogeneous sensing, when the
optical wavelength works at 1560nm, and the refractive index of the core and cladding layer is 1.56 and 1.49, respectively.

As described in section 3.1.3, the $k$ vector along the radial direction within the microresonator structure is neglected, when the waveguide based sensitivity is derived. Therefore, the actual $n_{\text{eff}}$ measured by experiments is smaller than the calculated based on this theory. However, this waveguide based theory gives specific formula to derive the sensor sensitivities. Therefore, it is easier to decide the relationship between the sensitivity and the factors of the sensor affecting it. In contrast, using conformal transformation method, introduced in section 3.1.4, includes the $k$ vector along the radial direction. This gives more accurate solutions for calculating sensitivities. Moreover, besides the sensitivity calculation, it can be used to calculate the resonant wavelength and FSR, as well. However, approximations need to be used to solve Eqn. 3.12. This introduces inaccuracy into the solutions. However, the decreasing trend of the surface in Fig. 3.4, with the increasing waveguide height, is the same as Fig. 3.2. These two theories will be used to analyze the experimental data from Chapter 4.
Figure 3.4 Overall sensitivity plotted as a function of W and $R_c$ for TE fundamental modes based on conformal transformation method.

### 3.1.5 Finite-Difference Time-Domain (FDTD) Method

A common and useful numerical method for the design of microdisk/ring resonators is the finite difference time domain (FDTD) method.\textsuperscript{56-58} FDTD is a numerical method for solving time domain Maxwell’s equations.

In the FDTD method, Maxwell's equations can be numerically solved in software after discretizing the first two equations shown above. FDTD is an ideal simulation
method for the design of microdisk or microring resonators. And compared to analytical methods, it gives more accurate and direct results. However, the simulation method requires a huge amount of memory and operation time of a working station as the number of simulated points increases. FDTD method will be used to calculate the coupling efficiency from the dielectric waveguide to the thin film InGaAs photodetector in this thesis.

3.1.6 Detection Limit (DL) and Quality (Q) Factor

Sensitivity is an important factor used to evaluate a sensor. But this measure alone is not sufficient for quantitatively characterizing the performance of a sensor, since the function of a sensor is to identify the targeted material in the sample. The detection limit is another important parameter to evaluate the performance of a sensor. The detection limit, defined as the smallest detectable change of the sensor’s parameters caused by analytes, is directly related to the smallest analyte amount that a sensor can detect. In the homogeneous sensing with resonant-wavelength-shift scheme, the detection limit \( DL \propto d_{\lambda} \propto \frac{d\lambda_c}{S} \). (Eqn. 3.15)

Here, \( d\lambda_c \) represents the minimum distinguishable wavelength shift that the sensor is able to detect. The minimum detectable \( d\lambda_c \) is theoretically determined by system resolution and is independent on the device properties (such as Q factor or FSR). \(^{49}\)

Sensor resolution is one concept related to the detection limit, and this term takes into account the spectral resolution of the system and system noise. Many factors, such as
device sensitivity, spectral resolution, and system noise (e.g. temperature, vibration), contribute to the detection limit of the sensor. In the resonant-wavelength-shift scheme, the most popular method to monitor the spectral shift is to track the position of the extremum (i.e., minimum or maximum which depends on the measurement configuration). 59

Sensitivity alone could characterize the sensors’s performance for zero system noise. In the two measurement mechanisms described, amplitude and spectral noise contribute to the errors in determining the resonant mode positions. Amplitude noise refers to the cumulative noise added to the spectral mode profile. Noise sources include thermal and shot noise in the photodetector, laser relative intensity noise, and quantization error. In an optimized system, the extremum can be located quickly. With added amplitude noise, the actual extremum is not likely to be at the exact center frequency of the Lorentzian shape (as it should be). Thus, the amplitude noise results in a random spectral deviation of the measured spectral location of the resonant mode. Therefore, it is important to analyze this deviation and its dependence on the magnitude of the noise and the linewidth of the mode. 59

Because the system noise could result in misidentification of the extremum, there exists the possibility for other points within the optical bandwidth of the resonant modes to be identified as the extremum. Under this circumstance, a high quality factor (Q factor) resulting a sharp extremum, is preferred. The Q factor is defined as the ratio of a resonant wavelength to its resonant bandwidth, which equals to the full width at half maximum.
(FWHM) of the resonance. Therefore, the detection limit is proportional to FWHM/S or 1/(QS).\textsuperscript{49}

The Q factor plays an important role in evaluating the performance of an optical microresonator sensor. A higher Q factor generally leads to a lower detection limit. The Q factor is determined by the geometry of the microresonator and the power loss in the microcavity.

### 3.1.7 Free Spectral Range (FSR)

The free spectral range (FSR), is defined as the difference between two adjacent resonant wavelengths, is another important parameter of the sensors. For single mode microring resonators, a sufficiently large FSR provides a wider detection range. According to Chao’s paper, the FSR for each propagation mode is

\[
FSR = \frac{\lambda_c^2}{2\pi R_c \cdot (n_{\text{eff}} - \lambda_c \frac{\partial n_{\text{eff}}}{\partial \lambda_c})} = \frac{\lambda_c^2}{2\pi R_c \cdot n_g} \text{ } \text{Equation 3.16}
\]

Here, \( n_g \) is the group velocity. FSR can also be calculated by the conformal transformation method described in section 3.1.1.4.

High sensitivity and low detection limit are two issues to be considered in the design of optical microresonator sensors. Single mode microring resonator sensors are preferred, because single mode operation can eliminate transverse modes competition. For a multimode resonator, each mode can create its own periodic resonance, which can result in the resonances from different modes to being too close to distinguish. However,
smaller feature size of the sensor will increase the difficulty of fabrication. Thus, there is a tradeoff between device sizes and the fabrication simplicity.\textsuperscript{49}

In this thesis, two polymer materials SU8 and PMMA with 0.8 refractive index contrast are chosen as the core and cladding layer of the microresonators. According to the theories described in section 3.1.3 and 3.1.4, and the material property, a 2 µm thick waveguide was chosen to optimize the sensitivity. Based on the consideration of the detection range and its later integration with microfluidic systems, which will be described later, the radius of the microring is optimized and determined to be between 500 and 600 µm.

**3.2 Design of Digital Microfluidic Systems**

The technologies used for the design of EWD microfluidic devices have already been broadly investigated. EWD devices modify the contact angle of a fluid droplet by an electric field applied between a polar or conductive fluid and an electrode underneath a dielectric. The change in contact angle, when applied nonuniformly, results in a capillary force. When an adjacent electrode is turned on, if a portion of the droplet also overlaps a grounded electrode, the droplet meniscus is deformed asymmetrically and a pressure gradient is established between the ends of the droplet, which results in bulk flow towards the energized electrode. The EWD microfluidic system works with most electrolyte solutions, and does not allow direct ohmic current flow, which minimizes the heat generation and electrochemical reactions. Moreover, as stated previously, silicone oil used in an EWD system can prevent droplet evaporation, and EWD microfluidics is compatible with microscopy.\textsuperscript{60, 61}
In this thesis, the microfluidic design issue is how to realize the required microfluidic systems that are suitable for integration with optical sensors and that do not interfere with the optical functions of the sensors. There are several issues: First, a thin Teflon-AF layer was spincoated (~80nm) on both the top and bottom plates to provide a hydrophobic surface for smooth droplet movement; Second, Pryolene C was chosen as the dielectric layer because of its high dielectric constant (3.15 at 60Hz); Third, Chromium was used to fabricate metal electrodes because of its good electric conductivity and high corrosion resistance. The choices for the top plate and other factors of the microfluidic systems will be discussed later in section 3.3. silicone oil was used as ambient medium to prevent droplet evaporation and dispel air bubbles.

3.3 Design of Integrated Optical Sensing Systems

Although technologies for planar optical microresonator sensors and EWD based digital microfluidic systems have already been investigated and developed separately, the realization of an integrated optical microresonator sensing system integrated with a digital microfluidic system entails more than simply putting them together. The following analysis describes the issues considered when integrating the digital microfluidic systems with optical sensors.

3.3.1 Design of an Integrated Chemiluminescence Sensor in a Digital Microfluidics Platform

An integrated optical chemiluminescence sensing system based upon the heterogeneous integration of a compound thin film photodetector with an electrowetting-based coplanar microfluidics system was demonstrated as part of this thesis. The
microfluidics aspects of this work were performed collaboratively with Dr. Richard Fair’s group, and include the work of PhD student Randall Evans. Dr. Fair and Mr. Evans provided the microfluidic design and structures, and helped with the testing of the microfluidics systems. In this integrated chemiluminescence sensing system, two types of chemical solutions are dispensed and mixed to generate an orange chemiluminescent optical output. The optical signal generated from the chemiluminescent reaction were detected by the integrated photodetector. A thin film metal-semiconductor-metal photodetector (about 1.2 µm thick) was integrated onto the top plate of the microfluidic system in order to sense the optical signal without affecting the movement of the fluids, as shown in Fig.3.5. A glass top plate is chosen for patterning metal electrodes and integrating with thin film photodetector. The Teflon-AF layer on the top plate was coated after the photodetector was integrated to protect the photodetector from interacting directly with the droplet. This demonstration of the integration and operation of an active optical device with a microfluidic system was the first demonstration of the integration of optical sensing devices with EWOD based digital microfluidic systems. The fabrication and test of this system is in Chapter 4 of this thesis.
Figure 3.5 Side view of a co-planar electrowetting chip integrated with a thin film photodetector. The top plate is customized and integrated with a photodetector by the author.²

3.3.2 Design of a Chip Scale Integrated Optical Microring/disk Sensor with a Digital Microfluidics System

The co-design of planar microresonator sensors and EWD microfluidic systems are critical to the realization, operation, and ultimate manufacturability of the integrated system. To co-integrate the sensors and microfluidics, the microfluidic system was designed to address the sensor with the fluidic analyte using a tapered, hydrophilic via. An acrylic plastic plate was chosen as the top plate, because it is easier to drill holes in it than in a glass top plate. Since the size of the microresonator diameter was no more than 600 µm, the size of the hole to be drilled in the top plate should be more than the microresonator diameter. A size of 1 millimeter was chosen herein. The size of the electrodes in the microfluidics was 0.8 mm by 0.8 mm, and the width of the microfluidic channel is 1 mm, which is the same size as the diameter of the hole. The thickness of the
The top plate was 500 µm; therefore, the volume of the droplet dispensed should be between 0.5 and 1 µL, in order for the droplet to be presented to the surface of the microresonator sensors. The diameter of the reservoir on the bottom plate of the microfluidic was 4 mm, which can be filled with 2 µL of liquid for dispensing. This design enabled the droplet to be actuated to the via location, and to extrude up into the via to address the optical microresonator sensor, as shown in Fig. 3.6. It also provided a consistent upper cladding index of refraction for the optical input/output optical waveguides (to/from the microresonator), since the dispensed analyte droplet extending into the via was confined to a constant area. In order to realize the coupling and detecting of optical signals in the microring sensor when it was integrated with the digital microfluidics system, both the input and output ports of the waveguides, which are vertically coupled with microresonators, were pigtailed with optical fibers.

Figure 3.6 Scheme of an optical microresonator sensor integrated with a digital microfluidic system.
3.3.3 Design of an Integrated Optical Microresonator Sensor with a Thin-film Semiconductor Photodetector and EWD Microfluidic system

The integration of a thin film photodetector with a polymer microring resonator has been reported. In has demonstrated a polymer microring sensor was integrated with an InGaAs based metal-semiconductor-metal (MSM) photodetector on a Si substrate for chip scale sensing applications. By monitoring the output photocurrent from the photodetector as a function of wavelength, the spectral response of the microring and its shifts were detected. In this thesis, this work will be extended through the integration of a microresonator sensor and a photodetector with an EWD digital microfluidic system. It is a big step toward the next generation of “Sensor-on-a-Chip” integrated systems.

In the design of the integrated microresonator/PD optical sensing system, which is to be integrated with an EWD digital microfluidic system, a thin film InGaAs MSM photodetector was heterogeneously integrated on top of a substrate. The structure layers of the InGaAs based material is listed in Table 3.1. Next, a single mode optical fiber is pigtailed to the input waveguide of microdisk and the electrical wires are glue with the metal electrode pads on the substrate. Finally, the pigtailed microdisk sensor integrated with photodetector will be attached to the top layer of microfluidics system, as is shown in Fig. 3.7a. After being integrated with the microfluidic system, this sensing system is finally able to detect glucose solutions in small droplets with different concentrations delivered by the microfluidic system. It is a significant step toward higher functionality optical system integration with a microfluidics platform. In the measurement, a tunable laser will be used to generate input optical signals, and the photocurrent of the thin film
InGaAs MSM photodetector will reflect its response to each wavelength output from the microdisk sensor.
Figure 3.7 (a) Side view of an integrated chemical optical microdisk sensor integrated with an electrowetting chip and photodetector; (b) Integrated microdisk sensor with thin film InGaAs MSM photodetector.

The power budget in this integrated sensing system is an important issue to be considered, and dictates the coupling considerations from the waveguide to the photodetector. The microresonator is addressed with a tunable laser. The maximum optical power coming from the tunable laser is 6.11 dBm. The attenuation loss due to the optical fiber can be neglected. The coupling loss from the single mode optical fiber from the laser to the input SU8 waveguide varies greatly in experiments. There are many factors that determine the coupling loss, including the alignment position between the fiber and waveguide, the optical properties of the epoxy used for pigtail the fiber to the input waveguide, and the structures and optical properties of the fiber and the waveguide. The coupling loss from the pigtailing fiber to the waveguide is unknown, and the coupling loss from the other parts of this system, which include the coupling loss from the waveguide to the microresonator, and the same from the resonator to the waveguide, is small compared to the former. The optical power coupled from the output waveguide to the photodetector is the next important design factor to be simulated. Here, in the FullWAVE module of the RSoft, Photonics Suite is used as simulation software. Three dimensional finite difference domain simulations were used to calculate how much optical power in the waveguide is coupled into the photodetector. In this simulation, a waveguide with a 5µm width is patterned on the top of the thin film photodetector. The
refractive indices of the waveguide and the photodetector at $\lambda = 1550\text{nm}$ are listed below in Table 3.2. A continuous wave laser source was excited and input, and two monitors were placed on the waveguide before and after the MSM photodetector. An illustration in Fig. 3.7(b) shows the structures and positions of the waveguide and photodetector. Using 3D FDTD theory, comparing the monitored power in the waveguide passing before and through the PD, the estimated power absorbed by the PD is 85%.

**Table 3.2: Refractive index of the materials used for waveguide and photodetector.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Refractive Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>SU8</td>
<td>1.57</td>
</tr>
<tr>
<td>InGaAs</td>
<td>$3.4^{62}$</td>
</tr>
<tr>
<td>PMMA (polymethyl methacrylate)</td>
<td>1.4893</td>
</tr>
<tr>
<td>SiO2</td>
<td>1.45</td>
</tr>
</tbody>
</table>
Chapter 4 Fabrication and Test

4.1 Fabrication and Test of Microresonators

4.1.1 Configurations of Microdisk Resonators

There were two different microdisk resonator configurations fabricated for this thesis, as shown in Fig. 4.1a and 4.1b. The arrows pointing to a line across the microresonator indicates the location of the corresponding cross sectional schematic shown at the left of each photomicrograph. Both microresonators are vertically coupled, as described in Chapter 2. The waveguide lies above top or below bottom the microresonator and not thus vertically coupled. The microdisk resonator shown in Fig. 4.1a is buried underneath the upper cladding layer, and the input/output waveguide is patterned on the top of the microresonator. The coupling is controlled by the thickness of the middle cladding layer, which is the polymer material spincoat PMMA. The thickness and optical properties (e.g. index of refraction) of the PMMA determines the coupling efficiency between the waveguide and the microdisk. This configuration is appropriate for communication component applications such as filters; however, this configuration is less appropriate for sensing applications, where surface customization of the microresonator is utilized. If the surface was customized with a material that responded to a target analyte or the surface of the microresonator is directly contacted with the target analyte, then the waveguide, which would also be affected by the surface
customization, would also be affected by the interaction Fig. 4.1b shows a cross section of a structure that has a buried input/output waveguide integrated partially below the microdisk. This configuration is useful for both communication component applications and for sensing applications, since the microresonator surface is accessible without exposing the input/output waveguide to surface customization. 

Figure 4.1 Schematic cross sections of the two types of microdisk resonators demonstrated within this thesis, and photomicrographs of each resonator (a) A schematic cross section and photomicrograph of a microdisk resonator with the input/output waveguide integrated on top of the microdisk structure; (b) A schematic cross section and photomicrograph of a microdisk resonator with the input/output waveguide integrated partially under the microdisk structure. 

45
4.1.2 Fabrication of Microdisk Resonators

To fabricate the integrated microdisk and waveguide samples, a Si substrate with 3 micron thick SiO$_2$ lower cladding layer was used for both the samples with the input/output waveguides above the microdisk, and for the samples with the waveguides embedded in/below the microdisk. For the integrated structure with the waveguide integrated above the microdisk, shown in Fig. 4.1a, the Si/SiO$_2$ substrate was first spin coated with a 2.2 µm thick layer of SU-8 2002, a photosensitive polymer that exhibits good optical transparency at input wavelengths longer than 600 nm, and has excellent chemical and mechanical stability. A 400 µm diameter microdisk was then formed by Reactive Ion Etching (RIE) the SU8 with a photoresist mask. A 0.2 µm thick layer of 495PMMA A4 was then coated on top of the entire sample, and served as the waveguide upper cladding layer and the separation layer between the microdisk and the input/output waveguide. Finally, a SU-8 2002 waveguide was spin coated and photolithographically defined (15 µm wide) so that it crossed the edge of the microdisk, as shown in the scanning electron micrograph (SEM) image in Fig. 4.2(a). In order to avoid undesired coupling from the input beam to the collection fiber, the output waveguide was curved to turn 90 degrees with a 5 mm radius of curvature. Fig. 4.2(a) shows the scanning electron micrograph image of microdisk curved edge which is buried underneath the straight waveguide
Here is the recipe for fabricating the microdisk structure in Figure 4.1.1a:

1. Prepare SiO$_2$/Si wafer with a piranha etch (H$_2$SO$_4$:H$_2$O$_2$ = 3:1)

2. Spin coat SU-8 2002;

3. Hard cure;

4. Spin coat PR4000A;

5. Pattern it with a microdisk mask

6. RIE etch away SU8 and then remove PR4000A photoresist;

7. Spin coat PMMA;
8. Post bake;

9. Spin coat SU-8 2002;

10. Soft bake;

11. Expose sample in 365 nm UV light with waveguide photomask;

12. Post-exposure bake and develop;

13. Hard cure.

Figure 4.2(b) Scanning electron micrograph image of the microdisk curved edge (left) with straight waveguide (right) buried underneath the microdisk.
The microdisk structure shown in Fig. 4.1b is a vertically coupled microdisk with the waveguide integrated underneath it. For most biological and chemical sensing applications, the microdisk should be exposed for surface customization, and exposing the input/output waveguide should be avoided. The fabrication process is similar to that described in Fig. 4.1a, but is inverted in layer order. First, a 2 µm thick SU-8 polymer waveguide layer was spin coated onto the SiO2/Si substrate. Next, a 0.2 µm PMMA separation layer was spin coated on top of the waveguide structure. Finally, a 500 µm microdisk in SU-8 2002 was patterned photolithographically so that the edge of the microdisk overlapped the input/output waveguide for coupling, as shown in the scanning electron micrograph (SEM) image in Fig. 4.2(b).

Here is the recipe for fabricating the microdisk structure in Figure 4.1b:

1. Prepare SiO2/Si wafer with a piranha etch (H2SO4:H2O2 = 3:1)
2. Spin coat SU-8 2002;
3. Soft bake;
4. Expose sample with a waveguide photomask (for the buried waveguide structure);
5. Post-exposure bake;
6. Develop in SU8 developer and rinse with isopropyl alcohol (IPA);
7. Hard cure;
8. Spin coat PMMA;
9. Post bake;
10. Repeat procedure 1-7 with a microdisk mask for exposure at procedure 4.
Since the waveguide is located above (or below) the microdisk, this is a vertically coupled microresonator, in contrast to laterally coupled microresonators, which are typically patterned, as discussed previously, by EBL or NIL. In the vertically coupled microdisk resonators shown in Fig. 4.1, the gap between the waveguide and the resonator is controlled by the spin coating process that dictates the separation/coupling layer thickness located between the microresonator and the waveguide. Coupling efficiency in these vertical microdisk structures mainly depends upon the polymers used (i.e., the indices of refraction), the microdisk diameter, the overlap between the optical waveguide and microdisk cavity, and propagation losses. Thickness control of the separation/cladding layer is available through spin coating (which depends upon the viscosity of the photoresists, spinning speed, etc.). In addition, based on the fabrication process, the cavity loss of the configuration in Fig. 4.1a is higher than that of the other structure in Fig. 4.1b, because its surface roughness is higher due to RIE etching.

To capture an image of the microdisk, the microdisk with the waveguide located above the microdisk was first tested with a pigtailed laser diode operating at a wavelength 660 nm. A tapped optical table and three-axis stages were used for the fiber-to-waveguide alignment. Fig. 4.3 is a photomicrograph of the microdisk shown in Fig. 4.1a with optical input at \( \lambda = 660 \) nm, with the optical path through the microdisk clearly observable as a ring of red light due to bending losses and scattering loss. The input/output waveguide, which is much less lossy, is less clearly visible at the top of the microresonator in the photomicrograph.
4.1.3 Measurement Results

Figure 4.3 Vertically coupled microdisk resonator with red light optical excitation at a wavelength of 660 nm. 63

The microdisk resonators were measured with an optical experimental setup which consisted of a linearly polarized broadband amplified spontaneous emission (ASE) light source, an optical erbium doped fiber amplifier (EDFA), and a polarization preserving optical fiber connected to the input port. A polarization preserving fiber and an optical spectrum analyzer (OSA) were used to record and analyze the microresonator output optical spectrum. Fig. 4.4 shows a schematic illustration of the experimental setup for measuring the output spectrum of a microdisk resonator.
To conduct the measurements, first, a broadband optical beam, which was generated by an ASE source from Lightwaves 2020 Inc., was amplified by a commercial erbium doped amplifier (EDFA, Lightwaves 2020 Inc.). In order to measure clear spectral output from the microdisk sample, the broadband optical beam (after being amplified) was linearly polarized with a half wave plate linear polarizer in free space. This optical beam was then end-face coupled into the input waveguide, which was vertically coupled to a microdisk resonator. The output light was collected by an end-face-coupled multimode fiber (50/125 μm) at the end of the output waveguide, which was then connected to an ANDO AQ-6315E OSA. The output spectrum of the microdisk resonator was then measured by OSA. 63
Both structures of the vertically coupled microdisk resonators shown in Fig. 4.1 were tested using the same methodology described above. Fig. 4.5 shows typical measured output spectra for the two types of vertical microdisk resonator structures excited with an input wavelength range of 1545 nm-1555 nm. As anticipated, multiple resonant peaks are observed at the output in both cases. Fig. 4.5a shows the measured output spectrum of the microdisk resonator structure shown in Fig. 4.1a. The measured free spectral range (FSR) is about 1.2 nm, and the Q factor is about 5000 for wavelengths at approximately 1550 nm. FSR is defined as the separation (typically measured at a pair of maxima or minima) between two adjacent resonant wavelengths. Fig. 4.5b shows the measured output spectrum of the microdisk resonator shown in Fig. 4.1b. The measured FSR of the resonances is about 1 nm and the Q factor is about 4560 for wavelengths at approximately 1550 nm. Thus, the functional characteristics of these two structures are very similar, and there is no detriment to the structure noted by embedding the waveguide inside/underneath the microresonator. The different FSRs result from the different dimensions of the microdisk resonators (500 µm-diameter microdisk in Fig. 4.1(a) vs. 600 µm-diameter microdisk in Fig. 4.1(b)).
Figure 4.5(a) Measured output spectrum of a microdisk structure with the microdisk on the bottom and the waveguide on top, corresponding to the structure in Fig. 4.1a; (b) Measured output spectrum of a microdisk structure with the microdisk on top with a buried waveguide, corresponding to the structure in Fig. 4.1b. 63
4.2 Fabrication and Test of Digital Microfluidic systems

An EWD digital microfluidic system concludes electrodes on a the bottom and a top plate. To fabricate the electrowetting microfluidic systems, Cr electrodes were first deposited and patterned on a glass substrate. An 80 μm SU8 gasket was next patterned, followed by a coating of parylene and teflon. In order to monitor the movement of the droplets, the bottom plate was fabricated on a transparent glass substrate. The top acrylic plastic plate was coated with indium tin oxide (ITO) and Teflon.

The process for fabricating the bottom plate and top plate.

Fabrication process for bottom plate:
1. Prepare glass substrate with a piranha etch;
2. Deposit Cr (300nm) by electron-beam evaporation;
3. Using photoresist as an etch mask to etch Cr electrodes;
4. Evaporate a 3 μm dielectric layer of Parylene C;
5. Coat 100nm thick of Teflon AF;
6. Open contact area.

The fabrication process for the top-plate (Acrylic plastic plate) is:
1. Sputter 100 nm of a transparent conducting layer of ITO;
2. Coat a hydrophobic layer of Teflon AF;
3. Drill reservoir holes and the via (only for the microresonator sensing).

The cross section and a photomicrograph of an EWD based digital microfluidic system is shown below in Fig.4.6. Also, Fig 4.7 is a series of photographs showing how
the dyed water droplets were actuated and dispensed from the reservoir, and were moved and mixed along the channel.

Figure 4.6 Cross section and a photomicrograph of an EWD based digital microfluidic system.
Figure 4.7 Series of photographs showing how the dyed water droplets were actuated and dispensed from the reservoir, and were moved and mixed along the channel.

4.3 Fabrication and Test of Thin Film MSM Photodetectors

The material used to fabricate the I-MSM photodetectors used in this work was grown by molecular beam epitaxy on an InP substrate. The grown layers consisted of a 40 nm InAlAs (cap layer), 20 nm InGaAlAs (superlattice graded layer), 800 nm InGaAs (absorbing layer), 200 nm InAlAs (cladding window layer), and a 200 nm InGaAs...
(selective etch stop layer), with all of the layers nominally undoped, as listed in Table 4.1. Metal fingers (Ti/30nm-Pt/40nm-Au/200nm) were defined with a lift off process. The photoresist was first patterned on the InGasAs material by standard photolithography and then metal Ti/Pt/Au was deposited on top using metal evaporation. Using lift-off, the metal fingers were patterned on the substrates, as shown in the photomicrograph in Fig. 4.8a. The next step was mesa etching with H₂O₂/citric acid (1:10). The etched MSM mesas were then coated with Apiezon W and immersed in HCl, which selectively removed the InP substrate (Fig. 4.8b). Next, the MSM devices were bonded to a transparent transfer diaphragm, and the Apiezon W was removed with TCE (shown in Fig. 4.8c). Finally, the MSM devices were transferred (and, in that transfer, inverted) and bonded to the host substrate metal pads (Ti/Au), which were deposited onto the host substrate. The pads on the photodetector and the pads on the host substrate formed metal/metal bonds, as shown in Figures 4.8d. The MSM photodetector shown in Figure 4.8 was used in the integrated system in section 4.4.1.²

Table 4.1: Material structures used for large-area high-speed I-MSMs grown by Dr Brown’s group.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>InAlAs (cap layer)</td>
<td>40 nm</td>
</tr>
<tr>
<td>Layer Description</td>
<td>Thickness</td>
</tr>
<tr>
<td>-------------------------------------------------------</td>
<td>-----------</td>
</tr>
<tr>
<td>InGaAlAs (Superlattice grade)</td>
<td>20 nm</td>
</tr>
<tr>
<td>InGaAs (Absorption layer)</td>
<td>80 nm</td>
</tr>
<tr>
<td>InAlAs (Supporting layer)</td>
<td>20 nm</td>
</tr>
<tr>
<td>InGaAs (Etch stop layer)</td>
<td>20 nm</td>
</tr>
<tr>
<td>InP (substrate)</td>
<td>50 um</td>
</tr>
</tbody>
</table>

The responsivity of the I-MSM was measured using a calibrated pigtailed laser diode at 660 nm, and an optical power meter. The typical measured responsivity for surface normal illumination of the I-MSM (on the side without fingers) was 0.39 A/W.²
Figure 4.8 (a) Top view of MSM photodetector metal fingers deposited on the InGaAs substrate (b) Top view of MSM photodetector metal fingers after mesa etching; (c) MSM thin film devices after substrate removal (viewed from the backside): these thin film devices are 1 micron thick, and the metallized finger/pad surface shown in Figure 1 is on the other side of the devices; (d) Thin film MSM photodetector on a transfer diaphragm.  

4.4 Fabrication and Test of Integrated Chip Scale Sensing Systems

4.4.1 Integrated Chemiluminescence Sensor in a Digital Microfluidic Platform

4.4.1.1 Fabrication and System Integration

The chemiluminescence sensor integrated with a digital microfluidic system consisted of two parts: a thin-film photodetector and a coplanar EWD-based microfluidic system. Fig.3.5 shows the cross section of a coplanar electrowetting-based microfluidic system integrated with a thin film photodetector. In the coplanar digital microfluidic system, which was fabricated by Advanced Liquid Logic in a printed circuit board (PCB) process, the gap between the top plate and the surface of the chip was approximately 90 μm. The chip was attached to an electrical controller, which was connected and manipulated by a computer controlled graphical user interface. Electrical voltages applied to the chip to actuate the liquid droplets were about 220 V. The InGaAs-based photodetectors were grown, fabricated, and then heterogeneously integrated onto a 35
mm by 50 mm glass substrate using metal/metal bonding, and then this entire integrated structure was attached to the electrowetting system.

The fabrication of the thin film photodetector has been described in section 4.3. To complete the integration of the I-MSM PD to the microfluidic system, the photodetector was mounted on one side of a glass substrate. The microfluidic system must present hydrophobic surface to the droplets contained therein, also as shown in Fig. 3.5. To realize this hydrophobic surface, a 1 µm thick Teflon AF layer was coated on top of the photodetector and glass plate. Glass plate was used as the top plate of the digital microfluidic system, and the host substrate where the thin film InGaAs photodetector was integrated to, as well. The photodetector (and the glass plate it was integrated onto) were mounted as the top plate, with the photodetector facing downward, and it was aligned with the linear array of electrodes on the lower plate of the microfluidic system. All biases and data from the photodetector were applied and measured, respectively, using a Keithley source measurement unit (SMU), which was connected to the photodetector via two probes connected to the contact pads that protruded from the top glass plate.

When pyrogallol and NaOH solution are mixed with one part of 30% H₂O₂, Trautz-Schorigin reaction occurs and an orange chemiluminescence light is emitted. The intensity of the light decays with time after mixing. The optical signal generated from this chemoluminescent reaction was detected by the integrated photodetector. Both solutions are immiscible in silicone oil, thus silicone oil were used to enhance droplet movement. The chemicals used in the experiments were dispensed from on-chip reservoirs, as shown in Fig. 4.9.
A series of measurements were performed to demonstrate the detection of an optical signal from the mixing of two fluids that produce chemiluminescence. The first experiment was conducted with and without integrating with the EWD microfluidic system to compare the chemiluminescent signals detected by the photodetector. First, two droplets were mixed on a Teflon AF-coated glass cover slip, 0.13-0.17 mm thick (not the acrylic substrate onto which the photodetector was integrated), that was placed right over
the photodetector. The chemiluminescent optical signal was measured by the integrated thin film I-MSM photodetector, by the side that had no metal fingers (which is the side that faced the chemiluminescent droplet in the electrowetting system in the next experiment). The mixture on the cover slip was 2.5 µl of H₂O₂ mixed into 5 µl of pyrogallol solution. The data was taken for about 210 seconds during the entire mixing process, and used as baseline for comparison to the measurement results with the integrated sensing PD/microfluidic system. Ascend measurement was performed by using the integrated photodetector and microfluidic system. A droplet of the pyrogallol solution was first actuated and moved to a location underneath the photodetector, and a smaller droplet of the H₂O₂ was next moved and mixed with it. The resulting photocurrents from the thin-film I-MSM with 1V MSM bias voltage were measured with a Keithley 236 SMU, and are displayed in Fig. 4.10. Fig. 4.10 shows the comparisons of the photocurrents measured by the PD with and without integrating with the EWD digital microfluidic system. The measured optical peaks detected by the photodetector, with and without integrating with the EWD digital microfluidic system generated by the chemiluminescence reaction, agree well. It shows that the optical photodetector sensor and the digital microfluidic system are able to cooperate together. The EWD microfluidic system is able to deliver droplets to where the sensor is located programmably, and the optical sensor can detect the chemiluminescent signal generated by the mixed droplets.
Figure 4.10 Comparisons of measured photocurrent by photodetector when droplet mixed with/without microfluidic system. \(^2\)

The second measurement also used the integrated electrowetting/photodetector system shown in Fig. 4.9. Two droplets of H\(_2\)O\(_2\) and pyrogallol solutions were individually actuated and mixed underneath the optical sensor to generate a chemiluminescent signal, the resultant mixed droplet was then moved relative to the photodetector, and the photodetector output current sensed the changing optical signal with the changing droplet position, as shown in Fig. 4.11.

The integrated thin film photodetector was placed facing downward with the fingerless photodetector face toward the microfluidic channel. A 1.2 mm thick Teflon AF-coated acrylic top plate was placed between the photodetector and the droplets. The
H$_2$O$_2$ and pyrogallol solutions were dispensed from their reservoirs separately, and were actuated to mix together. The droplet was mixed near the photodetector, then moved under the photodetector, then out from under the photodetector, then moved under the photodetector and away from the photodetector again, using actuation voltages. Fig. 4.11 shows the measured current from the photodetector for this optically emitting droplet movement. After being moved under the photodetector twice, the droplet was then moved far away from the photodetector, and this is reflected in the response of the photodetector, as shown in Fig. 4.11. The optical photocurrents increase to local maximum at 60 and 175 seconds, when the mixed droplet was moved underneath the photodetector, as shown in Fig. 4.11. The optical photocurrent then decreases as a function of time (after approximately 60 s) due to the droplet being transported away from the photodetector. Also, it decreases after approximately 175 s due to both the decay of the optical output intensity of the Chemiluminescence, since the droplet was located underneath the photodetector.
Figure 4.11 Chemiluminescent optical signal sensed by the thin film photodetector integrated with the electrowetting system. Two droplets were first dispensed from their reservoirs, mixed together, then moved under the photodetector. The chemiluminescent droplet was next moved away from the photodetector, moved back under the photodetector again, and then, finally, moved away from the photodetector. The photodetector output current reflects this optically chemiluminescent droplet movement. 2

The chemiluminescent signals detected by the thin film photodetector are shown in the data in Figs. 4.10 and 4.11. As expected in both cases, when the pyrogallol and H₂O₂ solutions were mixed, the initial optical output intensity was large, and then decayed as a function of time. The second peak of the photocurrent from the mixed droplets in the electrowetting system, shown in Fig. 4.11, was slightly lower than the first
one in magnitude due to the signal decay. The movement velocity of the mixed droplet was slow because of the high viscosity of the droplet, and due to the large droplet size.

The two experiments conducted above demonstrated how an optical sensor integrated with an EWD based digital microfluidic system works. The InGaAs-based thin-film photodetector was heterogeneously integrated onto a glass substrate, which was then integrated with the digital microfluidic system. This integrated thin-film I-MSM photodetector was able to detect the chemiluminescent signals generated by a pyrogallol solution mixed with H₂O₂, both external to, and internal to, the electrowetting microfluidics system. This demonstration of the heterogeneous integration of an active thin-film compound semiconductor optical device with a digital microfluidics platform is the first step toward a fully integrated chip scale optical sensing system integrated with digital microfluidics. Applications for these systems include biological and chemical sensing for medical and environmental sensing and monitoring.

4.4.2 Optical Microresonator Sensors Integrated with EWD Digital Microfluidic Systems

4.4.2.1 Fabrication and System Integration

The design of the integrated optical microresonator sensing systems with EWD digital microfluidic system discussed in Chapter 3 was fabricated and tested for this thesis. The microresonator sensor fabrication began with the deposition of the input/throughput and drop waveguides on a SiO₂/Si substrate. The 4 µm thick SiO₂ acted as an optical lower cladding layer for the microresonator waveguides. First, the SiO₂/Si substrate was coated with a 2 µm thick photosensitive SU8 2002 layer that was patterned
into channel waveguides. A ~170 nm thick layer of 495 PMMA, which has a lower refractive index than SU8, was then spin coated on top of the sample, and served as the waveguide upper cladding layer and the coupling layer between the microring and the waveguides. Finally, a 600 µm diameter microring was defined in another top layer of SU8, as shown in Figure 4.12a. To fabricate the microdisk sensors, instead of defining a microring in the top layer SU8, a 500 µm diameter microdisk was defined instead.

To fabricate the electrowetting microfluidic system, Cr electrodes were first deposited and patterned on a glass substrate. An 80 µm SU8 gasket was next patterned, followed by a coating of Parylene and Teflon. The acrylic top plate (1 mm thick) was coated with an optically transparent conductor, indium tin oxide (ITO), and then Teflon, and subsequently a 1 mm diameter via was mechanically drilled to allow the actuated fluid to address the optical sensor, as shown in Figure 4.12c. To push water droplets up into the via to address the polymer microresonator sensor, this via was tapered and intentionally hydrophilic (Teflon did not coat the inside of the via). Figure 4.12d is a photomicrograph of the microfluidic system with this top plate via addressed with a water droplet that contains red dye (to provide visual contrast). This droplet was actuated to the via by the microfluidic system shown in Figure 4.12d, with the water/dye droplet extruded from the via. In the integrated system, the microresonator sensor was located in this via.

To optically address the microring sensor when it was integrated with the digital microfluidic system, the input port and drop ports of the waveguides were pigtailed with single mode and multimode optical fibers, respectively. Each fiber was pigtailed to the
sample on one end, and had a commercial FC connector on the other end of the fiber. The setup used for fiber pigtailing is shown in Fig. 4.13. Norland Optical Adhesive 65 was used to attach the fiber to the waveguide. The refractive index of this optical adhesive after UV curing is around 1.524. The group effective refractive index of this adhesive at 1550nm is 1.4682, which is close to the optical fiber and the polymer SU8. The optical microresonator sensing system will be flipped over and integrated with the digital microfluidic system, making the optical alignment from the fiber to the waveguide extremely difficult. Therefore, a fiber pigtail interface was necessary to optically connect the integrated optical system to the optical test equipment. Figure 4.12b is a photograph of an optical microring sensor integrated onto Si/ SiO$_2$ that has been adhesively bonded to a glass carrier and fiber pigtailed. The sensor is on the surface of the Si/ SiO$_2$ structure (facing the reader). This optical sensor structure was then inverted and bonded to the microfluidic system such that the microresonator was centered at the top of the via in the microfluidic system. Figure 4.12e is a photograph of this integrated chip-scale optical microresonator sensor/microfluidic system.
Figure 4.12 The optical microresonator sensor integrated with a digital electrowetting microfluidic system: (a) photomicrograph of the 600 µm diameter microring resonator with the input and drop (output) waveguides; (b) photograph of the optical microring sensor bonded to a glass carrier and pigtailed (external optical input/output fibers attached to the integrated sensor for testing); (c) photograph of the microfluidic system with which the microresonator sensor was integrated (photo before integration); (d) photomicrograph of the microfluidic system addressing the top plate via with a water/red dye droplet; the droplet was actuated from the reservoir to the via position, and subsequently extruded from the via; (e) photograph of the optical microring resonator sensor (shown in Figure 4.12a) integrated with the digital electrowetting microfluidic system (shown in Figure 4.12c).
Figure 4.13 Setup used for fiber pigtailing the optical fiber to the microresonator sensors.

4.4.2.2 Experimental Measurements

The fabricated microring and microdisk resonator sensors were both integrated with (separate) digital microfluidic systems, and the operation of each of these chip-scale
integrated systems was tested using glucose to vary the index of refraction at the sensor surface. These two sensor systems were also tested with two different spectral measurement systems, as illustrated in Figure 4.14. The first experimental apparatus, shown in Figure 4.14a, uses a broadband amplified spontaneous emission (ASE) input source and an optical spectrum analyzer (OSA) at the output. This experimental setup evaluates spectral intervals as small as 5 picometers (pm); however, a spectral interval of 10 pm was used to minimize noise (the OSA reading was too noisy at the 5 pm resolution). The second experimental test apparatus, illustrated in Figure 4.14b, utilizes a tunable laser source and a photodetector. The laser has a larger optical output per spectral interval (5 pm for the laser/PD system) than the ASE, which results in a better SNR than the ASE/OSA system.

Figure 4.14(a) Spectral measurement setup for the integrated microdisk sensor/microfluidic system: an optical broadband amplified spontaneous emission source and an optical spectrum analyzer.
The ASE/OSA measurement method was used for the integrated microdisk resonator/microfluidic sensing system. First, the broadband ASE source was coupled into an erbium-doped fiber amplifier (EDFA, Lightwaves 2020 Inc.) to amplify the input optical signal. After it was linearly polarized with a half wave plate, this optical signal was coupled into the single mode optical fiber that was pigtailed to the microdisk resonator input waveguide. The light that was not vertically coupled to the resonator was measured as the output from the throughput waveguide. This output waveguide was pigtailed to a multimode optical fiber, which was routed to the OSA. Labview was used to automate the experiment; the output spectra from the OSA were recorded every 10 seconds.

To measure the performance of the microring resonator integrated with the EWD microfluidic system, the tunable laser/photodetector measurement apparatus was used. The pigtailed microring sensor was optically addressed with a HP81618A tunable diode
laser (single mode output). Labview was used to control the tunable laser to sweep the output wavelength from 1520-1570 nm in 10 pm increments. The entire optical spectrum sweep was 10 seconds. The optical signal from the tunable diode laser was input to the EDFA to amplify the laser signal. A half-wave plate was used to control the input laser polarization to the integrated sensor system, and the optical signal was delivered to the microresonator sensor through a single mode optical fiber. The output optical signal from the integrated microring/microfluidics system was collected by a pigtailed multimode fiber at the output of the drop waveguide, which was then routed to a HP81623A optical detector. The output spectrum of the microring sensor was continuously and automatically recorded by a computer using Labview every time the tunable laser completed a full spectral sweep (every 10 seconds).

To test the integrated microresonator sensor/microfluidic systems, test droplets were dispensed and actuated by voltage control of each electrode on the digital microfluidic system. A controller board in an associated computer dictated the on/off switching of the electrodes, which manipulated the droplet movements, including dispensing and translation. Droplets were first dispensed from the on-board reservoir, actuated from metal pad to metal pad using applied voltages, and thus were moved from the reservoir to the position where the via and microresonator sensor were located. When the droplets reached the via, the hydrophilic nature of the via inner walls caused the droplet to push up into the via to address the microresonator sensor located at the top of the via.
The integrated microresonator/microfluidics system was characterized through the shifts in the resonance spectra of the microresonators due to the change in refractive index at the surface of the sensor caused by the presentation of the glucose solution. The shifts are measured in nm of wavelength shift per refractive index unit (RIU). Actuating and measuring the spectra for deionized water provided the baseline spectral data for the microresonator sensors. First, water was dispensed from the on-board reservoir and presented to the sensor through the via. Next, the reservoir was filled with glucose, which was actuated droplet by droplet to the sensor, where the droplets mixed with the previously dispensed water, thus generating different concentrations of glucose solution as droplets were added. The glucose concentrations, and thus, indices of refraction, were estimated using the volume of droplets dispensed and the known starting volume of deionized water presented to the microresonator through the via. These changes in glucose concentration presented a change in index of refraction to the microresonator sensor, which appeared as shifts in resonant peaks in the output spectrum.

The 500 μm diameter microdisk sensor throughput waveguide was monitored due to the low optical SNR in the ASE/OSA measurement system. The throughput port offers higher intensity output, but less sharp resonance to monitor. The 600 μm diameter microring sensor was monitored through the drop port, which was enabled through the use of the tunable laser/photodetector measurement system, which had a higher input optical power. Resonant wavelengths appear as dips in the spectrum observed at a throughput port (for the microdisk sensor), and as peaks in the spectrum observed at a (optional) drop port (for the microring sensor). These resonances can be analyzed to
determine the shift in resonant wavelength that correlates to a change in index of refraction at the resonator sensor surface.\textsuperscript{64}

Figure 4.15(a) Measured spectral shift of the vertically coupled microdisk resonator sensor as a function of index of refraction (through glucose concentration) presented to the sensor surface. Solid line (Pt. A) de-ionized water with refractive index \(~1.3330\); long-dashed line (B) 1.36 g/100ml glucose solution with refractive index at \(~1.3349\); short-dashed line (C) 3.46 g/100ml glucose solution with refractive index \(~1.3378\); long/short dashed line (D) 4.29 g/100ml glucose solution with refractive index \(~1.339\).
Figure 4.15(b) Resonant wavelength shifts in the microdisk sensor for different concentrations of D-glucose in DI water.

The measured spectral shifts in the 500 µm diameter microdisk sensor at the throughput port as a function of index of refraction change at the sensor surface (through varying glucose concentration) are shown in Fig. 4.15(a). Each data point is the average of six spectra measured for the indicated glucose concentration. The baseline, water, is spectrum A, and subsequent dispensing and actuation of glucose droplets for mixing at the via (B, C, D, in order), show a consistent spectral shift with increasing glucose concentration.
The six measured resonances (all wavelength peaks from 1553-1559 nm) used to calculate the data points were also used to calculate the error bars. The error bars are +/- one standard deviation for the same wavelength peaks as the data, from 1553-1559 nm. The horizontal error bars were calculated based upon the uncertainty in the volume of each droplet dispensed. Thus, as more droplets were dispensed, the uncertainty in the volume of the droplet increased, thus increasing the uncertainty of the droplet glucose concentration. Note that the volume uncertainty in the dispensed glucose droplets also contributed to the error bar calculation of the glucose concentration. The measurement uncertainty (i.e. error bar) in the vertical direction (resonant wavelength shift) could arise from noise from the scanning resolution of the optical spectrum analyzer (0.05 nm), noise from polarization changes due to fiber motion (external to the integrated system), and the accuracy of the glucose concentration (the uncertainty in the volume of the total sum of the dispensed droplets accumulated increased as more droplets were dispensed). As is shown in Fig. 4.15(b), the average resonant wavelength peak shifts rise approximately linearly with the increase in index of refraction (i.e. glucose concentration; the index of refraction of glucose is in Reference 65). The response of the fabricated microdisk sensor to the change index of refraction at the resonator surface was measured to be approximately 95 nm/RIU for this sensor. Thus, a chip scale optical microresonator sensor integrated with a digital microfluidics system was demonstrated herein. Glucose solution of different concentrations were dispensed, actuated, and sensed on this lab-on-chip platform.
To test the 600 µm diameter microring resonator sensor integrated with EWD microfluidics, the laser/photodetector apparatus was controlled by Labview, and the measured spectral resonance data was imported into Matlab for more advanced data analysis. The port offers higher intensity output, but less sharp resonances to monitor. The 600 µm diameter microring sensor was monitored through the drop waveguide, which offers higher intensity output than the drop port, since the resonant peaks are monitored. Resonant wavelengths appear as peaks in the spectrum observed at a drop port (for the microring sensor). These resonances can be analyzed to determine the shift of resonant wavelength that correlates to a change in index of refraction at the sensor surface.

The wavelength shift could be calculated by taking the difference in wavelength of the initial (deionized water) baseline and the resonant spectral peak position after each glucose presentation. The measured spectral shifts in the 500 µm diameter microdisk sensor at the drop port as a function of index of refraction change at the sensor surface (through varying glucose concentration) are shown in Fig. 4.16(a). Each data point is the average of five spectra measured for the indicated glucose concentration. The baseline, water, is spectrum A, and subsequent dispensing and actuation of glucose droplets for mixing at the via (B, C, D, in order), show a consistent spectral shift with increasing glucose concentration.

The five measured resonances (all wavelength peaks from 1560-1565 nm) used to calculate the data points were also used to calculate the error bars. The error bars are +/- one standard deviation for the same wavelength peaks as the data, from 1560-1565
nm. The horizontal error bars were calculated based upon the uncertainty in the volume of each droplet dispensed. Thus, as more droplets were dispensed, the uncertainty in the volume of the droplet increased, thus increasing the uncertainty of the droplet glucose concentration. The measurement uncertainty (i.e. error bar) in the vertical direction (resonant wavelength shift) could arise from noise from polarization changes due to fiber motion (external to the integrated system), and the accuracy of the glucose concentration (the uncertainty in the volume of the total sum of the dispensed droplets accumulated increased as more droplets were dispensed).

As is shown in Fig. 4.16(b), the average resonant wavelength peak shifts rise approximately linearly with the increase in index of refraction. The response of the fabricated microdisk sensor to the change index of refraction at the resonator surface was measured to be 87 nm/RIU for this sensor. This response will be compared to the theoretical expectation in Chapter 5.
Figure 4.16(a) Measured spectral shift of the vertically coupled microring resonator sensor as a function of index of refraction (through glucose concentration) presented to the sensor surface. Solid line (Pt. A) de-ionized water with refractive index \(~1.3330\); long-dashed line (B) 0.2 g/100ml glucose solution with refractive index at \(~1.3338\); short-dashed line (C) 1 g/100ml glucose solution with refractive index \(~1.3344\); long/short dashed line (D) 2.45 g/100ml glucose solution with refractive index \(~1.3364\).
Figure 4.16(b) Resonant wavelength shifts in the microring sensor for different concentrations of D-glucose in DI water.

In these measurements, a chip scale optical microring sensor integrated with a digital microfluidic system was demonstrated. Glucose solutions of different concentrations were dispensed, actuated, and sensed on this lab-on-chip platform. The response of the fabricated microring sensor to the change index of refraction was measured to be 87 nm/RIU for this sensor, which is slightly lower than the integrated microdisk sensor. It is probably due to the errors in this analysis resulting from changes
in relative power coupling between closely-spaced transverse modes that can cause apparent shifts in the resonant wavelength, or small polarization changes in the input beam, which can produce changes in the resonant wavelengths.

Thus, herein, two integrated systems that integrate chip-scale microresonator sensors (microdisks and microrings) with EWD microfluidic systems are discussed herein. These integrated optical sensor/microfluidic systems were designed, fabricated, and successfully tested using varying index of refraction at the sensor surface as presented by glucose solutions of different concentrations. The glucose was dispensed, actuated, and presented to the integrated optical sensor by the EWD microfluidics systems. The index of refraction changes in the microresonator sensors due to the presentation of these different glucose concentrations resulted in an optical resonance spectral shift that was measured and characterized for both systems. The integrated microdisk resonator sensor demonstrated a sensitivity of 95 nm/RIU, and the integrated microring resonator a sensitivity of 87 nm/RIU. These two chip-scale systems, which integrate optical sensors with electrowetting microfluidics systems, are a first step toward chip-scale, low power, fully portable integrated sensing systems for medical diagnostics and environmental monitoring.

**4.4.3 Chip Scale Integrated Optical Microresonator Sensors with Embedded Thin-Film Photodetector on EWD Digital Microfluidics Platforms**

**4.4.3.1 Fabrications and System Integration**

The design of a planar optical sensing system, consisting of an optical microdisk resonator and a thin film InGaAs photodetector, integrated with an EWD digital
microfluidic system, was fabricated and tested for this thesis. This sensing system included optical detection with a chip scale integrated sensing system. This integration of a sensor, and optical component, and a microfluidic system is a significant further step toward chip-scale, low power, fully portable integrated sensing systems for medical diagnostics and environmental monitoring.

To fabricate the optical microresonator/PD/EWD sensing system, an InGaAs based thin film MSM photodetector and an optical microresonator sensor were fabricated and integrated onto a SiO₂/Si substrate. First, the metal electrodes (Pt/Ti) were deposited on the SiO₂/Si substrate. Next, a thin film MSM PD was fabricated separately (see section 4.3), and then transferred and bonded to the metal pads patterned on the SiO₂/Si substrate. The photodetector bond was annealed by heating at 180°C for 20 minutes for mechanical strength. The fingers of the photodetector were 2 µm by 30 µm, the space between the fingers was 3 µm, as shown in Fig. 4.17(c). The total area is 110 µm by 245 µm, with 100 µm by 150 µm contact area, and 35 µm by 100 µm sensing area. The SU8 waveguide was next patterned on top of the photodetector and the SiO₂/Si substrate. Finally, a 2.2 µm microresonator was patterned by the procedure described in section 4.3, and is shown in Fig 4.17(a). The fabrication process of the electrowetting microfluidic system is the same as described in section 4.2.
Figure 4.17 Photomicrographs (a and b) and schematic (c) of the optical microresonator sensor integrated with a MSM thin film photodetector: (a) photomicrograph of the 600 µm diameter microdisk resonator with the input and drop (output) waveguides; (b) photograph of the MSM thin film photodetector embedded in an optical waveguide; (c) schematic figure which shows the dimensions of the photodetector features.
To address the microresonator sensor and the MSM PD when the optical system was integrated with the digital microfluidic system, the input port of the optical waveguide was pigtailed with a single mode optical fiber using optical adhesive (see section 4.4.2.1), and the metal pads which contacted the PD were connected to the copper electrical wires using tapes. Figure 4.17(b) is a photograph of an optical microdisk sensor integrated onto Si/SiO$_2$ that has been adhesively bonded to a glass carrier and fiber pigtailed. The sensor is on the surface of the Si/SiO$_2$ structure, as shown in Figure 4.17(a). This optical sensor structure was then inverted and bonded to the microfluidic system such that the microresonator was centered at the top of the via in the microfluidic system.
Figure 4.18 Photographs of the optical microresonator sensor integrated with a thin film InGaAs photodetector; (a) Top view of the chip, with a 600 µm diameter microdisk resonator integrated with a thin film photodetector; (b) Photograph of the optical microring sensor bonded to a glass carrier and pigtailed. The external optical input fiber and output electronic wires are attached to the integrated system for testing.
4.4.3.2 Experimental Measurements

The fabricated microdisk resonator sensor and photodetector were integrated with a digital microfluidic system, and the operation of this integrated system was tested using glucose to vary the index of refraction at the microresonator sensor surface. This integrated optical biosensor system was tested with the spectral measurement systems, as shown in Fig. 4.19.

Figure 4.19 Spectral measurement setup for the integrated microdisk sensor/microfluidic system: a tunable diode laser and a current/voltage measurement part. Blue lines mean optical connections, and black are electrical connections.

To measure the performance of the microdisk resonator integrated with the EWD microfluidic system, the tunable laser apparatus was used. The input port of the pigtailed microdisk sensor was optically addressed with a HP 81618A tunable diode laser (single mode output), and the output port of the sensor was sensed by the integrated photodetector, which was biased with a Keithley 236 Source Measurement Unit. Labview
was used to control the tunable laser to sweep the input wavelength from 1540-1560 nm in 20 pm increments, and the Keithley 236 Source Measurement Unit to record the photocurrent output at 1-10 V voltage biases. The entire optical spectrum sweep was 240 seconds. When the tunable laser tuned to a certain wavelength, it gave an output trigger signal, which was received and taken as the input trigger by the Keithley current measurement unit. After the input trigger, the current measurement unit would measure the photocurrent. When finishing the current measurement, the Keithley unit would generate an output trigger, and this trigger would be sent to the tunable laser and taken as the input trigger of the laser. Then the laser would switch to next wavelength, and so on.

The optical signal from the tunable diode laser was input to the EDFA to amplify the input laser signal. A half-wave plate was used to control the input laser polarization to the integrated sensor system, and the optical signal was thus delivered to the input single mode optical fiber. The optical signal in the output waveguide was sensed by the thin film photodetector, which was embedded in the optical waveguide. The output spectrum of the microdisk sensor was continuously and automatically recorded by a computer using Labview every time the tunable laser completed a full spectral sweep.

To test the integrated microdisk sensor/PD/microfluidic system, test droplets were dispensed and actuated by a voltage (70 V amplitude) control of each electrode on the digital microfluidic system. A controller board in a computer dictated the on/off switching of the electrodes, which manipulated the droplets, including dispensing and translation. Droplets were first dispensed from the on-board reservoir, actuated from metal pad to metal pad using applied voltages, and thus were moved from the reservoir to
the position where the via and microresonator sensor were located. When the droplets reached the via, the hydrophilic nature of the via inner walls caused the droplet to push up into the via to address the microresonator sensor at the top of the via.

Actuating and measuring the spectra for deionized water provided the baseline spectral data for the microresonator sensor. First, water was dispensed from the on-board reservoir and presented to the sensor through the via. Next, the reservoir was filled with glucose, which was actuated droplet by droplet to the sensor, where the droplets mixed with the previously dispensed water, thus generating different concentrations of glucose solution as droplets were added. The glucose concentrations, and thus, indices of refraction, were estimated using the volume of droplets dispensed and the known starting volume of deionized water presented to the microresonator through the via. These changes in glucose concentration resulted in a change in index of refraction at the microresonator sensor, which appeared as shifts in the resonant peaks in the output spectrum.

4.4.3.3 Measurement Results and Data Analysis

The measured spectral shifts in the 600 μm diameter microdisk sensor at the throughput port as a function of index of refraction change at the sensor surface (through varying glucose concentration) are shown in Fig. 4.20. Each data point is the average of five spectra measured for the indicated glucose concentration. The baseline, water, is spectrum A, and subsequent dispensing and actuation of glucose droplets for mixing at the via (B, C, D, in order), show a consistent spectral shift with increasing glucose concentration.
Figure 4.20(a) Measured spectra shift of the vertically coupled microdisk resonator sensor as a function of index of refraction (through glucose concentration) presented to the sensor surface. Solid line (Pt. A) de-ionized water; long-dashed line (B) 0.33 g/100ml glucose solution; short-dashed line (C) 1 g/100 ml glucose; long/short dashed line (D) 1.8 g/100 ml glucose solution.
After identifying the peaks, the change in the peak position as a function of time was tracked as different glucose concentrations (i.e. different indices of refraction) were presented to the sensor. The wavelength shift was calculated by taking the difference in wavelength of the initial (deionized water) baseline and the resonant spectral peak position after each glucose presentation. Five peaks were measured and averaged to produce the data point in Figure 4.20. The five measured resonances (all wavelength
peaks from 1543.5-1547.5 nm) used to calculate the data points were also used to calculate the error bars. The error bars are +/- one standard deviation for the same wavelength peaks as the data, from 1543.5-1547.5 nm. The horizontal error bars were calculated based upon the uncertainty in the volume of each droplet dispensed. Thus, as more droplets were dispensed, the uncertainty in the volume of the droplet increased, thus increasing the uncertainty of the droplet glucose concentration. Note that the volume uncertainty in the dispensed glucose droplets also contributed to the error bar calculation of the glucose concentration. The measurement uncertainty (i.e. error bar) in the vertical direction (resonant wavelength shift) could arise from noise from polarization changes due to fiber motion (external to the integrated system), and the accuracy of the glucose concentration (the uncertainty in the volume of the total sum of the dispensed droplets accumulated increased as more droplets were dispensed).

As is shown in Fig. 4.20(b), the average resonant wavelength peak shifts rise approximately linearly with the increase in the glucose concentration, which linearly corresponds to the index changes of refraction of glucose. The response of the fabricated microdisk sensor to the change index of refraction at the resonator surface was measured to be 69 nm/RIU for this sensor. Because the height of the SU8 microdisk resonator is 2.2 µm, which is larger than the microresonators fabricated in section 4.4.2, the sensitivity measured is lower compared to the two sensing systems described in section 4.4.2.

The fabrication and test of a chip scale microdisk/PD optical sensing system integrated with an EWD digital microfluidic system was described in this section. A thin film InGaAs MSM photodetector was heterogeneously integrated on a Si substrate and
with the output polymer waveguide of a microdisk resonator. After being integrated with the microfluidics system, this sensing system was able to detect changes in index of refraction as presented by glucose solutions in small droplets with different concentrations, which were delivered by the microfluidics system.

In conclusion, the fabrication processes and the corresponding measurement results for three chip scale integrated sensing systems were detailed in this chapter. The goal for realizing the chip scale integration of optical microresonator sensors, waveguides and thin film InGaAs MSM photodetectors with EWD digital microfluidic systems has been reached. First, an integrated optical sensor based upon the heterogeneous integration of an InGaAs thin film photodetector with a digital microfluidic system was fabricated and tested. This demonstration of the integration and operation of an active optical device with a microfluidic system is the first step toward the integration of entire optical sensing systems with microfluidic systems. Second, chip scale optical microdisk/ring sensors integrated with digital microfluidic systems were demonstrated. It is a first step toward chip-scale, low power, fully portable integrated sensing systems. Finally, a chip scale sensing system, which is composed of a planar integrated optical microdisk resonator/PD sensing system and a digital microfluidic system, was realized. It is also a significant step toward higher functionality optical system integration with a microfluidics platform for medical diagnostics and environmental monitoring.
Chapter 5 Experiment vs. Theory

5.1 Characterization of Microresonators and Comparison to Experiments

For a microresonator sensor, the output field is equal to the input field multiplied by the loss factor between the coupling sections and the phase change. Therefore, the normalized output power for an orthogonal microdisk resonator (as is shown in Fig. 2.1), is

\[ I_{out} = \left| \frac{E_{2,2}}{E_{1,1}} \right|^2 = \left| \frac{k_1 k_2 a^{0.25} \exp(j0.25\phi)}{t_1 t_2 a^{0.5} \exp(j\phi) - 1} \right|^2 \]

(Equ. 5.1)

Here, \( a \) is the cavity loss factor and \( a = \exp(\alpha L/2) \), where \( \alpha \) is the attenuation coefficient and \( L \) is the round trip optical path length. \( k_1, k_2 \) are the cross-coupling coefficients between the waveguides (throughput and drop) and the microdisk cavity. \( t_1 \) is the transmission coefficient of the field passing through the coupling region in the throughput waveguide, \( t_2 \) is the transmission coefficient inside the microdisk cavity, and \( \phi \) is the round-trip phase shift. \( k \) and \( t \) are related by \( |k|^2 + |t|^2 = 1 \) for lossless coupling.

Assuming the structure of the coupling regions between the microdisk cavity and the waveguide is completely symmetric, then \( k_1 = k_2 = k \), \( t_1 = t_2 = t \).

The output spectrum of the microresonators can be calculated through the formula Equ. 5.1 and the conformal transformation method described in section 3.1.4. The calculations include two parts: the resonance profile and the resonant wavelength. The periodic resonance profile in the output spectrum can be theoretically calculated by Equ.
5.1, and compared to the measured microdisk resonator output. The coupling coefficient $t$ varies with the coupling geometry and the materials used. It can be derived using coupled mode theory\textsuperscript{54,66} or FDTD simulations. The parameters can also be extracted from fitting Equ. 5.1 to the experimental data. For SU-8 rib waveguides, the average attenuation coefficients of energy are $\alpha_{TE}$ and $\alpha_{TM}$, with respective values $\alpha_{TE} = 0.32/cm$ and $\alpha_{TM} = 0.456/cm$.\textsuperscript{67} Using this analytical approach, the shape and location of the microresonator resonances can be calculated.

The experimental results analyzed first were from the measurement of a chip scale optical microring sensor integrated with a digital microfluidics system, described in section 4.4.2. The calculated resonant wavelength spectrum (water baseline shown in Fig.4.16) is shown in Fig. 5.1 as dotted line. The resonant peaks from 1560 nm to 1564 nm (maxima points in the output spectrum) of the microring resonator were calculated based on the conformal transformation method described in section 3.1.4. First, the refractive index along z direction was $n_z$ calculated based on the Equ.3.8. $n_v$ was next derived based on $n_z$, which is also the effective refractive index $n_{eff}$. The resonant wavelengths were finally determined by $n_v$ and are shown in Table 5.1. In this analysis, the coupling coefficient $t$ was determined using the TE loss parameter given above with and a curve fit to Equ. 5.1 and found to be 0.35.

The theoretically calculated resonances are offset slightly from the experimentally measured resonances (Fig. 5.1), likely because the measured modes do not correspond to
the fundamental TE mode, which was used in the conformal transformation analysis. The agreement between theory and experiment is excellent for the spacing of the modes.

Table 5.1: The resonant peaks recorded from the output spectrum shown in Fig. 4.16 (DI water) and the comparisons to the resonant wavelength (between 1560-1565 nm) calculated by the conformal transformation method.

<table>
<thead>
<tr>
<th>Resonant Peaks Measured (nm)</th>
<th>Resonant Wavelength Calculated (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1560.75</td>
<td>1560.65</td>
</tr>
<tr>
<td>1561.58</td>
<td>1561.43</td>
</tr>
<tr>
<td>1562.38</td>
<td>1562.20</td>
</tr>
<tr>
<td>1563.17</td>
<td>1562.98</td>
</tr>
<tr>
<td>1563.97</td>
<td>1563.87</td>
</tr>
</tbody>
</table>
Figure 5.1 Normalized output spectrum of the optical microdisk resonator (the solid red line shows the measured output spectrum, and the dotted line shows the calculated data).

Figure 5.2 shows the measured transmission profile measured in the experiment described in section 4.4.3 and the simulated transmission profile for comparison. The experiment results are from the measurement of a chip scale optical microring/photodetector sensor integrated with a digital microfluidics system. The measured photocurrent, which shows the resonant wavelength spectrum (water base line shown in Fig.4.19) is normalized and shown in Fig. 5.2 as blue dotted line. The resonant
wavelengths from 1553nm to 1558nm (maxima points in the output spectrum) of the microdisk resonator were also calculated based on the conformal transformation method described in section 3.1.4. The resonant wavelengths are listed in Table 5.2. In this analysis, the fitted value of the coupling coefficient $t$ was 0.55.

Table 5.2: The resonant peaks recorded from the output spectrum shown in Fig. 4.19 (DI water) and the comparisons to the resonant wavelength calculated by the conformal transformation method.

<table>
<thead>
<tr>
<th>Resonant Peaks Measured (nm)</th>
<th>Resonant Wavelength Calculated (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1543.08</td>
<td>1543.11</td>
</tr>
<tr>
<td>1543.90</td>
<td>1543.93</td>
</tr>
<tr>
<td>1544.70</td>
<td>1544.75</td>
</tr>
<tr>
<td>1545.48</td>
<td>1545.57</td>
</tr>
<tr>
<td>1546.30</td>
<td>1546.39</td>
</tr>
<tr>
<td>1547.10</td>
<td>1547.19</td>
</tr>
<tr>
<td>1547.92</td>
<td>1548.08</td>
</tr>
<tr>
<td>1548.70</td>
<td>1548.82</td>
</tr>
</tbody>
</table>
Figure 5.2 Normalized output spectrum of the optical microdisk resonator (the solid red line shows the measured output spectrum, and the dotted blue line shows the calculated data).

Both the calculated and measured data are shown in Fig. 5.2 for comparison. The real resonant modes measured in the output spectrum include influences by many factors, such as intermodal coupling, and the presence of higher-order transverse modes. Here those effects can be neglected without losing the primary peak resonance, including the local secondary peaks of the higher order modes. A better agreement between the calculated results and experimental measurements is shown from Fig 5.2, compared to the results from Fig. 5.1. This is likely because the measured modes are the fundamental
TE mode calculated. Therefore, the theory described in Chapter 3 can help us to estimate the output spectrum of the microresonator sensors, e.g. resonant wavelength, FSR, and coupling coefficient in the microcavity coupling region.

5.2 Sensitivity of Microresonator Sensors and Comparison to Experiments

A chip scale optical microring sensor integrated with an EWD digital microfluidic system was demonstrated, as described in section 4.4.2. In addition, a chip scale optical microdisk resonator/PD sensing system integrated with a digital microfluidic system is further demonstrated in section 4.4.3 of this thesis. These chip scale integrated optical sensor/microfluidic systems were tested using varying index of refraction at the sensor surface as presented by glucose solutions of different concentrations. The glucose solutions were dispensed, actuated, and presented to the integrated optical sensor by the EWD microfluidic systems. The index of refraction changes in the microresonator sensors due to the presentation of these different glucose concentrations resulted in an optical resonance spectral shift that was measured and characterized for the systems. The integrated microring resonator with a 600 µm diameter demonstrated a sensitivity of 87 nm/RIU, and the sensitivity of the integrated microdisk/PD sensor with 600 µm diameter was 69 nm/RIU. The measured sensitivities of the microring (2 µm height) and the microdisk resonators (2.2 µm height), are shown in Fig. 5.3 and compared to the calculated sensitivities based on the theory described in section 3.1.3. The solid line in Fig. 5.3 shows the calculated sensitivities as a function of the height of the microresonators. The sensitivity decreases from 112.28 to 65.57 nm/RIU as the height
increases from 1.8 µm to 2.3 µm. The two stars show the measured sensitivities of the integrated microring sensor with 2 µm height and the integrated microdisk/PD sensor with 2.2 µm height. The two data point is the average of wavelength shifts for three resonant wavelength measured for the indicated glucose concentration. The three measured resonances used to calculate the data points were also used to calculate the error bars. The error bars are +/−σ for the same wavelength shifts as the data. The measured sensitivities agree well with the calculated sensitivities for TM00 modes. And the decreasing trend of the measured sensitivity as the increase of the microresonator height is consistent with the calculated trend. Theories described herein help to decide some important factors (e.g. waveguide height, microresonator diameter), which affect the sensitivities of the microresonator sensors. These factors can be optimized in the microresonator design to result in better system characteristics.
Figure 5.3 Comparisons of the calculated and measured sensitivities plotted as a function of the height of the waveguide for TM\textsubscript{00} modes (the blue stars represent the measured sensitivities of the microdisk and microring sensors, and the solid line shows the calculated).

The resonant wavelength shifts as a function of index of refraction changes at the microresonator sensor surface produced by varying the glucose concentration can be calculated by the conformal transformation theory described in section 3.1.4. As shown in Fig. 5.2, the resonant wavelength of the integrated microdisk/PD sensor can be
calculated. The relative wavelength shift of the resonance can also be derived when the refractive index of the cladding layer changes from 1.333 (water) to 1.3343 (1g/dL glucose solution). The dotted lines in Fig. 5.4 show the calculated resonant wavelength shift for a 600 µm diameter SU8 microdisk sensor with the refractive index change of $1.4 \times 10^{-3}$ (the dotted blue line is the water baseline, and the dotted red line is the 1g/dL glucose solution). Meanwhile, the measured spectral shift by the optical sensor demonstrated in section 4.4.3 is also shown in Fig. 5.4 as solid lines. The baseline, water, is drawn as a solid blue line, and the subsequent wavelength shift at 1wt%, produced by dispensing glucose droplets and mixing at the surface of the sensor, is shown as a solid red line. Here, the side lobes in the measured output spectrum are coming from the higher order whisper gallery modes. The wavelength shift and the corresponding resonant mode profiles were calculated and fit well to the experimental data, as shown in Fig. 5.4. Theories described herein help to estimate the resonance wavelength shifts when the index of refraction changes at the microresonator sensor surface by varying the glucose concentration.
Figure 5.4 Comparisons of the measured and calculated normalized output spectrum shifts of a chip scale integrated optical microdisk/PD sensor with 600 µm diameter: The solid line shows the measured spectrum shifts in the experiments from 0wt% water (blue line) to 1wt% (red line) glucose solution. And the dotted lines show the calculated spectrum.

5.3 Coupling Efficiency from Waveguide to PD and Comparison to Experiments

The power budget in this integrated sensing system is an important issue to be considered and dictates the coupling considerations from the waveguide to the photodetector. Three dimensional finite difference time domain simulation was used to
calculate the coupling efficient of optical power from the waveguide coupled into the photodetector. In the simulation, a waveguide with a 5µm width is patterned on the top of the thin film photodetector. The refractive indices of the waveguide and the photodetector at $\lambda = 1550\text{nm}$ are listed below in Table 3.2. A continuous source field analogous to that of a continuous wave laser source field was launched into the input facet of the waveguide, and two monitors were placed on the waveguide before and after the MSM photodetector, as shown in Fig. 5.5. The power absorbed by the PD is estimated by taking the difference in the measured power from the power monitors in the waveguide before and after passing through the PD region. According to the simulation, the estimated power absorbed by the PD radiated from the waveguide is 85% of the incident power in the waveguide.
Figure 5.5 Simulation results for the coupling loss from the waveguide to the photodetector with FDTD: the optical power monitored before and after the PD are shown on the left; the 3D structure layout of the waveguide and PD is shown on the right.

Using the PD efficiency from the simulation and the two experimental results measured in section 4.4.2 and 4.4.3, the power loss for the fiber pigtailed to the waveguide can be estimated. The optical sensing system demonstrated in section 4.4.2 was the 600 µm diameter microring resonator, which was fiber pigtailed for both input and output detection. The output waveguide was fiber pigtailed and connected to an
81623A optical detector. The optical sensing system demonstrated in section 4.4.3 was the 600 µm diameter microdisk resonator integrated with thin film PD, which has only one port fiber pigtailed for optical input. The input sources of the two systems were both addressed by a tunable laser with 6.11 dBm maximum output optical power.

Assuming the losses are comparable in both experiments, a rough estimate of the coupling loss from the waveguide to the pigtailed fiber can be made. These attenuations include the losses due to the optical fiber, which can be neglected, the attenuation loss passing through the spatial polarizer, the amplification through the EDFA, the coupling loss from the optical fiber to the input waveguide, and the coupling loss from the other parts of this system, including the coupling loss from the waveguide to the microresonator, and the same from the resonator to the waveguide, and the transmission loss through the waveguide. The average optical power detected by the 81623A optical detector in system one is -44 dBm. The measured photocurrent shown in Fig. 4.19 was $4 \times 10^{-6} A$. Using the responsivity of the photodetector of 0.39 A/W, the optical power detected by the photodetector was -19.9dBm ($1.026 \times 10^{-5} W$). The coupling efficiency from the waveguide to the photodetector can be calculated depending on the coupling loss from the waveguide to the pigtailed fiber. Typically, this coupling loss from the waveguide to the pigtailed fiber varies from experiment to experiment. There are many factors that determine the coupling loss, including the alignment position between the fiber and waveguide, the optical properties of the epoxy used for pigtailing the fiber to the input waveguide, and the structures and optical properties of the fiber and the waveguide. Table 5.3 shows the calculated coupling efficiency from the waveguide to the
photodetector as the coupling efficiency the waveguide to the pigtailed fiber varies from 0.5% to 2% (coupling loss varies from 23dB to 17dB).

Table 5.3: The calculated coupling efficiency from the waveguide to the PD as the variation of the coupling loss the waveguide to the pigtailed fiber.

<table>
<thead>
<tr>
<th>Coupling loss from WG to pigtailed single mode fiber</th>
<th>Coupling efficiency from WG to PD</th>
</tr>
</thead>
<tbody>
<tr>
<td>17dB</td>
<td>19.5%</td>
</tr>
<tr>
<td>18dB</td>
<td>24.6%</td>
</tr>
<tr>
<td>19dB</td>
<td>30.9%</td>
</tr>
<tr>
<td>20dB</td>
<td>38.9%</td>
</tr>
<tr>
<td>21dB</td>
<td>48.9%</td>
</tr>
<tr>
<td>22dB</td>
<td>61.7%</td>
</tr>
<tr>
<td>23dB</td>
<td>77.6%</td>
</tr>
<tr>
<td>24.8dB</td>
<td>85%</td>
</tr>
</tbody>
</table>

This calculation is useful for developing the power budget in the sensing system design, and underscores the importance of an integrated optical system, which can minimize these fiber pigtailing coupling losses.
Chapter 6 Conclusions and Future Work

6.1 Conclusions

This dissertation has explored how to design, build, and measure chip scale integrated optical sensing systems with EWD based digital microfluidic systems, focusing on optical microdisk/ring resonator sensors integrated with digital microfluidic systems. In comparison to typical continuous fluidic systems integrated with planar optoelectronic devices, these chip scale integrated planar optical sensing systems with EWD based digital microfluidic systems eliminates the usage of moving parts, such as pumps or valves, and also enables droplets in microliter volumes to be automatically manipulated and detected. The ultimate goal of this dissertation is progress toward the realization of chip-scale, low cost, and fully portable sensing systems that integrate optical sensing with digital microfluidics. The challenging task of integration of active photonic devices with a microfluidic system has been achieved using heterogeneous integration technology.

Three chip scale optical sensing systems integrated with digital microfluidic systems were detailed in this thesis. As a demonstration of chip scale integrated chemiluminescence sensing, an optical sensor based upon the heterogeneous integration of a thin film PD integrated with a digital microfluidic system was designed, fabricated and measured. This demonstration of the integration and operation of an active optical device with a microfluidic system is the first step toward the integration of entire optical sensing systems with microfluidic systems. Next was the first demonstration of a chip-scale optical microdisk/ring sensor integrated with a digital microfluidic system, which
were fabricated and measured. Finally, a chip scale sensing system, composed of a planar integrated optical microdisk resonator/PD sensing system and a digital microfluidic system, was demonstrated and tested. This chip scale integrated system was demonstrated by measuring output resonant wavelength shifts due to the concentration changes of the glucose solutions presented to the sensor surface through the digital microfluidic system. This is a significant step toward higher functionality optical system integration with a microfluidics platform for medical diagnostics and environmental monitoring.

The co-design of the planar optical sensing systems and EWD digital microfluidic systems are necessary to the realization, operation, and ultimate manufacturability of the integrated system. Heterogeneous integration technology enables the capability to integrate thin film active optical devices (e.g. thin film InGaAs MSM photodetectors) with planar optical microresonator sensors on Si substrates. The Si substrates with patterned optical microresonator sensors were then inverted and integrated onto the top plate of the digital microfluidic systems. To co-integrate the optical sensors and microfluidics, the digital microfluidic system was designed to address the sensor cavity with the fluidic analyte using a tapered, hydrophilic via. An acrylic plastic plate was chosen as the top plate, since it is easier to drill holes in it than in a glass top plate. The material used to fabricate the gasket of the microfluidic is the photosensitive polymer material SU8. For fabrication easiness, the gasket height is no more than 100 µm. Since the size of the microresonator diameter was designed no more than 600 µm, the size of the hole to be drilled in the top plate was chose as 1 millimeter, a little larger than the microresonator diameter. The size of the electrodes in the microfluidics was 0.8 mm by
0.8 mm, and the width of the microfluidic channel is 1 mm, which has the same size as the diameter of the hole. The thickness of the top plate was 500 µm, which results in the volume of the droplet dispensed to be between 0.5 and 1 µL, which is consistent with the size of the droplet necessary to be presented to the surface of the microresonator sensors to completely cover the sensor. The diameter of the reservoir on the bottom plate of the microfluidic was 4 mm, which can be filled with 2 µL of liquid for dispensing. This design enabled the droplet to be actuated to the via location, and to extrude up into the via to address the optical microresonator sensor, to cover the sensor. It also provided a consistent upper cladding index of refraction for the optical input/output optical waveguides (to/from the microresonator), since the dispensed analyte droplet extending into the via was confined to a constant area. In order to realize the coupling and detecting of optical signals in the microring sensor when it was integrated with the digital microfluidics system, both the input and output ports of the waveguides, which are vertically coupled with microresonators, were pigtailed with optical fibers.

The optical microring/disk sensing systems and the EWD digital microfluidic systems were codesigned, and important parameters of these two systems were considered together and tradeoffs were analyzed for the system integration. Parameters of the digital microfluidic systems (such as metal electrode size, gasket height, dielectric layer material, containing volume of the reservoir) were selected to enable the operation of optical sensors that were 500 microns in diameter. Thus, the microfluidic and optical sensor system were designed to work together from a size perspective. The important factors affecting system sensitivities were theoretically analyzed to guide the sensing
system design. From the waveguide based theory and conformal transformation method described in Chapter 3, the specification of the microresonator sensors, which includes the resonant spectrums and sensitivities, was discussed. These calculations can help to identify suitable ranges for the height and diameter of the microresonator sensors for integration with the microfluidic system. This facilitates the design and the optimization of the microresonators integrated with digital microfluidics for sensing applications.

6.2 Future Work

There are a number of areas in which further work and improvement of the integrated sensing systems is possible. This includes the structure of the microresonator itself, as well as the system integration methods.

There are several suggestions to improve the sensitivity and detect of limit of microresonator sensors. In order to sense multiple targets simultaneously on one chip, or to offer a higher level of discrimination using multiple fused sensor signals, two or more microresonators can be integrated on one host substrate in close proximity. Differential sensing is critical in noisy environments. The configuration, fabrication process and material of two identical microresonator sensors, using one as a reference and the other as a sensor, noise from the environment can be cancelled by comparing the resonant wavelength shift between the two sensors.

Research into further integration of chip scale integrated photonic devices have been recently published, and may lead to further system integration. For example, a thin film edge emitting laser has been integrated with an independently optimized photodetector, both on a Si host substrate and interconnected with a polymer waveguide.
Therefore, it may be possible to heterogeneously integrate thin film InGaAsP/InP edge-emitting lasers (EEL), optical waveguides, microresonator sensors and photodetectors to realize fully integrated chip-scale optical sensing systems, and thereafter, to integrate these optical sensing systems with digital microfluidic systems.

The results of this thesis indicate that there is promise for the realization of highly sensitive, miniaturized, and portable photonic sensing systems integrated with digital microfluidic systems for applications in medicine, environmental sensing, and security.
References


62. http://www.batop.de/informations/n_InGaAs.html#


Biography

Lin Luan received the B.S. degree in Electrical Engineering from Shandong University, China, in 2001, and the M.E. degree in Communication and Information System, in 2004, from Peking University, China. She is pursuing the Ph.D. degree in Department of Electrical and Computer Engineering at Duke University. Her research interest includes photonic biochemical microresonator sensors integrated with digital microfluidic systems.

PUBLICATIONS


