A New Platform for Integrated Photonic Sensing with Nanostructured Thin Films

by

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A thesis submitted to
the Faculty of Graduate Studies and Research
in partial fulfillment of the requirements for the degree of
Doctor of Philosophy

Ottawa-Carleton Institute for Electrical and Computer Engineering
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ISBN: 978-0-494-52063-5
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ISBN: 978-0-494-52063-5

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Abstract

A new platform for optical sensing based on evanescent field interferometric devices and the application of dielectric nanostructured thin films as sensing environment is developed. Validation of this new platform is achieved through a complete cycle of design, fabrication and optical characterization. During the course of fabrication work, an integrated circuit compatible process for Si_3N_4 low temperature plasma etching is established. Optical characterization of Al_2O_3 structured thin films deposited on tilted substrates addressed two main aspects of film optical properties: the refractive index as related to deposition parameters and film birefringence. The dielectric films analyzed induce significant asymmetry with refractive index values that enable a retarder-like behavior. Quantitative analysis of the effect under normal incidence light demonstrated a strong orientation dependent transmittance with a large polarization rotation angle of $4.76^\circ/\mu\text{m}$. The enhanced rotation angle achieved presents an attractive and quite inexpensive technique for fabricating optical retardation plates of different dimensions.

The application of modal analysis on highly birefringent deposited TiO_2 films allowed determination of film anisotropy via refractive index evaluation. It is concluded that the refractive indices for TE and TM modes propagating along the film are different, with a $\Delta n=0.044$ obtained. Light propagating parallel to the substrate, as opposed to the normal propagation occurring in conventional applications, demonstrates a strong modal interaction, with transmitted light highly

affected by the column orientation. Limited interaction of the evanescent field with the birefringent cladding is compensated by the long propagating distance.

Optical loss analysis on fabricated waveguides shows that as the wavelength approaches $\lambda=1.62\text{ }\mu\text{m}$, attenuation due to intrinsic material absorption and scattering are reduced substantially, reaching 0.62 dB/cm. Finally, the optical characterization of interferometer-based fabricated devices with ZrO_2 or TiO_2 films deposited on the sensing area has demonstrated optical sensing capability of this platform. A maximum phase shift of $\pi/10$ on the MZI transmission spectra permitted the refractive index measurement of a known gas. In addition, device sensitivity is confirmed by measuring the output time response, where devices with TiO_2 film in sensing layer demonstrated higher sensitivity.

Dedicated to the memory of my mother

Jeta Celo

Acknowledgments

I gratefully acknowledge the role played by many people in completing this thesis. My special thanks to my thesis supervisors, Dr. Tom Smy and Dr. Jacques Albert for their guidance, continuous support and patience. I would like to express gratitude to P. Waldron, E. Post and S. Janz at NRC for the support with fabrication and optical characterization. Thanks also to M. Summers and M. Brett at University of Alberta for their support with GLAD film deposition. The financial support of Carleton University and Natural Sciences and Engineering Research Council of Canada that made possible this work is gratefully acknowledged.

Special thanks to staff and faculty in the Department of Electronics: Dr. G. Tarr, Dr. N. Tait, R. Vandusen and K. Adams for their assistance and advice during fabrication process; J. Lemieux, S. Bruce and N. Mikhail for providing technical support; A. Laronche for assistance with optical measurements; B. Power, P. Piccolo and S. Beekmans for being always there to assist. I also would like to thank and acknowledge my colleges: S. McGarry, A. Froimovitch, P. Chyurlia, B. Tamer, G. Romo and K.P. Yap.

Finally, I would like to recognize the unwavering support of my family. I would never have been able to complete this work without the encouragement of my wife Valbona. She has been a constant source of advice, inspiration and understanding. Thanks also to my sons Erdit and Patrick. They continuously reminded me of the important things in life.

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Nomenclature

List of Symbols

a	Measure of waveguide asymmetry
α	Flux angle of incidence
A_0	Amplitude of optical signal
α	Attenuation coefficient
b	Normalized guide index
β	Column inclination angle
c	Speed of light
$Chisq$	Variance of the model
E	Amplitude of electric field
ϵ_0	Permittivity of free space
f	Frequency
FL	Fresnel loss

j	$\sqrt{-1}$
I	Intensity of light
k	Proapagation constant
k_0	Free-space wave number
λ	Wavelength
λ_0	Wavelength of light in free space
L_{int}	Interaction length
$\mathcal{M}$	Characteristic matrix of optical model
μ_0	Permeability of free space
n	Refractive index
N_{eff}	Effective refractive index
n_{SL}	Refractive index in sensing layer
ω	Angular frequency
ϕ	Phase angle of transfer function
P_{in}	Optical power at input
P_{out}	Optical power at output
q_s	Reduction factor
R^2	Coefficient of determination
T	Optical transmittance

t	Time
θ	Birefringence angle
Φ	Azimuthal angle
V	Normalized frequency
v_d	Volume fraction of deposited material
v_p	Volume fraction of pore content
w	Rib width of a waveguide
Y_0	Optical admittances of air
Y_s	Optical admittances of substrate

List of Acronyms

Al_2O_3	Aluminum oxide
APCVD	Atmospheric pressure chemical vapor deposition
CO_2	Carbon dioxide
CVD	Chemical vapor deposition
CHF_3	Trifluoromethane
ECR	Electron cyclotron resonance
GLAD	Glancing angle deposition
HF	Hydrofluoric acid
IC	Integrated circuit
IO	Integrated optics
IOS	Integrated optical sensor
IR	Infra red
MZI	Mach-Zehnder interferometer
N_2	Nitrogen
N_2O	Nitrous oxide
NH_3	Amonia
NIR	Near infra red

NRC	National Research Council
O ₂	Oxygen
PECVD	Plasma-enhanced chemical vapor deposition
PM	Polarization maintaining
PR	Photoresist
PVD	Physical vapor deposition
RIE	Reactive ion etch
SCCM	Standard cubic centimeter per minute
SEM	Scanning electron microscope
SF ₆	Sulfur hexafluoride
Si ₃ N ₄	Silicon nitride
SiO ₂	Silicon oxide
SOI	Silicon-on-Insulator
SiH ₄	Silane gas
TC	Thermocouple
TE	Transmission electric
TiO ₂	Titanium oxide
TM	Transmission magnetic
ZrO ₂	Zirconium oxide

Chapter 1

Introduction

1.1 Motivation

During the last decade, a need for faster and improved means of detection, communication, and data processing has encouraged rapid growth in the field of all-optical components. This has led to the development of a variety of new sensing optical devices able to detect, measure, and process data using electromagnetic energy. In addition, materials technology and fabrication methods, particularly for semiconductors, have advanced accordingly, allowing devices to be built with fine structural details. As a result, all these developments have brought the technology to a point that new applications are required, where all-optical sensing components and circuits are replacing the conventional mechanical and electrical sensors.

In part, the motivation behind research and development activities in the optical sensing relies on the expectation that properties of optical sensing field are significantly advantageous over conventional sensing. Some of the advantages of optical over non-optical sensors are enhanced sensitivity, wide dynamic range, electrical passivity, insensitivity to electromagnetic interference, and multiplexing

capabilities. In this context, interferometric homogenous sensing [1] possess all these advantages. Operation of these devices is based on the interaction of the evanescent part of the propagating optical field with the measurand environment. In other words, the evanescent tail of the modal field travels into the sensing region filled with the measurand material and probes the optical properties of this environment. While some good sensitivities are reported, [2] there are limiting factors inherent to the existing methodology of device design and fabrication. A large refractive index difference at the waveguide core-measurand boundary causes: (a) the mode propagating into the waveguide to be strictly confined inside the core region, resulting in a very small evanescent field portion, and (b) the evanescent field traveling into the sensing area experiences high absorption and scattering losses. In addition, a large refractive index difference at the waveguide cladding-measurand boundary increases Fresnel reflection losses at the boundaries, and might also cause some interference of the reflected power with the evanescent field.

A possible solution to this problem would be a sensing environment with strictly controlled refractive index, porosity, structural configuration and high surface area to volume ratio. Finding a thin film structure able to overcome these limits becomes a challenging task in both aspects: optical waveguide design and fabrication technology. [3] From the existing technologies, such films can be deposited with a glancing angle deposition technique (GLAD), [4] which offers a high degree of control over the film nanostructure. A strictly controlled refractive index permits the evanescent field to be drawn deep into the sensing region. The ability to control the film configuration allows scattering losses to be reduced; this can be achieved by maintaining nanomaterial dimensions below the $\lambda/12$ limit. A

high surface area to volume ratio enhances the evanescent field-measurand interaction. As a result, all these factors contribute to enhance the optical sensitivity of such devices.

Furthermore, due to the large fragmentation of the sensor market and relatively small number of specific sensors that are needed generally, it is not economical to develop a special optical sensor for each individual application; instead one has to focus on development of so-called "sensor platforms" [1] which, with minor modifications, can be tailored to a large variety of applications. Having said that, the designing and fabrication processes described in this project address this issue. Dielectric material nanostructured thin films as a sensing environment represent a new platform for highly efficient optical sensing. By changing very few deposition parameters such as flux incidence angle or angular velocity of substrate rotation, a broad range of nanostructured films specific to individual applications can be created.

Another application driven motivation of this project is the optical characterization of highly anisotropic thin films. Optical materials with anisotropic electromagnetic (EM) response are essential elements in polarizing optics applications such as linear polarizers, prisms, and waveplates. These components are typically constructed from precisely cut naturally occurring uniaxial and biaxial crystals to change the relative phase between two components of polarization. E-beam evaporated dielectric material nanostructured thin films fabricated in this project are optically biaxial with the birefringence available for light propagating normal to the substrate. They present an inexpensive alternative to naturally occurring anisotropic materials which are generally incompatible with integrated photonic technologies. Due to its simplicity this technology may become very attractive for mass production of optical components.

1.2 Contributions

As outlined in the motivation section, the main objective that generated this work was to develop a new optical sensing platform based on interferometric sensing and dielectric material nanostructured thin films. With very small adjustments, this sensing platform can be tailored to specific applications. In view of the objective that generated this research work, the following contributions have been made:

1. In search for optical devices able to demonstrate the new concept of optically sensitive nanostructured layer, a new design concept [5] was presented. Formulation of a first order optimization technique for interferometric sensing led to the design of a multi mode interferometer coupler for optical sensing.
2. A modeling and design methodology was developed [6] to evaluate dielectric structured thin film growth parameters. As a result, evaluation of microstructural growth orientation, film density and refractive index, as well as film anisotropy were achieved. These parameters provided settings for further simulation of phase shift induced in Mach-Zehnder interferometer (MZI) output as a result of the refractive index change in the sensing area.
3. A new technique for silicon nitride (Si_3N_4) low temperature plasma etching [7] was established. Compatibility with integrated circuit process and the application of a carbon free etching based on SF_6/O_2 fluorine chemistry mixture in electron cyclotron resonance plasma source makes this method attractive to a wide range of applications such as integrated optics and micro electro mechanical system (MEMS) devices as well.
4. Aluminum oxide (Al_2O_3) high birefringent films were fabricated. Measurement of birefringence on fabricated films with light propagating normal to the substrate plane demonstrated strong orientation dependant transmitted

light; a film layer of thickness 840 nm achieved a 5° polarization rotation angle with the optical power at the output oscillating from 0 to 130 μW .

5. Birefringence properties of cladding layer titanium oxide (TiO_2) films were determined via modal analysis. Refractive index for both TE and TM modes was calculated, revealing a refractive index difference $\Delta n=0.044$.
6. Design, fabrication, and optical characterization of interferometer-based devices with zirconium oxide (ZrO_2) and TiO_2 films deposited on the sensing area was achieved. [8] Results obtained demonstrated the optical sensing capability of such devices through the refractive index measurement of a known gas.

1.3 Integrated Optical Sensing

Integrated optical sensors (IOS) are devices capable of measuring the presence, identity, or concentration of (bio) chemical compounds in different environments. Most IOS present an integration of a detection mechanism which generates a signal that corresponds to the concentration of the chemical compound measured with the optical read-out system. In these sensors, a change in the concentration of a chemical is converted into changes of the optical properties of a sensitive material, affecting the intensity and/or phase of an optical beam propagating into a waveguide medium. These parameter changes are detected by the optical system able to measure and correlate changes in the sensor's optical properties as a result of changes in chemical concentration.

A rapid development on the IOS was initially inspired by the success of optical fibre sensors. In later stages, growth of this field was driven by the aggressive

developments of the integrated optical devices and components for telecommunication applications, gaining momentum and taking advantage of all technological achievements in the area. At present, it is the combination of the optical waveguide with a multi-function electronic micro-system integrated in the same chip that makes IOS so appealing for a large range of applications. [9] The integrated optics approach allows for significant simplification of the optical system, since the light routing and processing can be accomplished by a waveguide circuit.

Besides the high degree of integration, there are other evident advantages of IOS: (a) the use of optical fibres allows for simple guidance of light towards and from the optical chip permitting remote sensing and combination of sensors in arrays or other configuration arrangements, (b) due to their immunity to electromagnetic and radioactive fields they can be used in very harsh environments, (c) there are a considerable number of technologies available for IOS fabrication, (d) all the design and simulation tools developed for optical telecommunication applications can be applied to IOS, reducing the design cost, and (e) this technology is unique in that kinetic experiments can be performed with relative ease using small amounts of material, and requiring no labeling of the reactants.

1.4 Evanescent field interferometric sensing

Techniques for recognizing the presence of the target vary for different IOS; however, there are a few optical parameters which the sensor in turn can detect and measure, with the refractive index and the absorption or luminescent properties being the most relevant ones affected by the chemical concentration. A change of refractive index expresses itself as a change of two parameters: the real part of the effective refractive index (N_{eff}) and the field profile, and the imaginary part of N_{eff} , which can be seen as a change of the absorption coefficient and

hence the modal attenuation. The effects of the measurand-induced changes on modal parameters depend on both, the change of optical properties of the sensing material and also on the fraction of the modal power which propagates through the waveguide and more specifically in the layer located in the evanescent wave region of the waveguide. Based on this phenomenon, the evanescent field sensing techniques have been applied in different applications. [1]

1.4.1 Sensing mechanism - surface versus homogenous

Essentially, the evanescent field sensing technique is based on a chemically sensitive material applied as a cladding layer within an opening obtained by locally removing the original (non-sensitive) cladding layer. [10], [11] The sensitive material is the medium where the evanescent field of the mode propagates. This configuration has several advantages. First, it allows the realization of general IO sensing platforms which later on can be provided with a specific cladding, enabling the capture of the target units aimed at a specific application. Secondly, the response time and diffusion paths of target molecules can be controlled by technological and geometrical means in the fabrication/deposition process of chemo-optical cladding layer. In this way the sensing layer can be tailored such that an acceptable response time can be achieved, even in the case of large target molecules. Thirdly, the amount of power overlap between the evanescent field and the sensing layer can be controlled in two ways: by changing the thickness of the sensing layer, [12] and/or by changing the length of the active region. By doing this, the effects on the light beam properties are much larger than in the case of a thin layer interrogated by a perpendicularly incident free space beam, where the interaction length would be equal to the thickness of the sensitive layer.

With respect to the thickness of the sensitive cladding layer two extremes can

be defined. The first one is commonly called the surface sensing; it has a very thin sensitive cladding layer, in the order of few nanometer and sometimes even thinner on the order of a monomolecular layer. In a good approximation, the modal field is constant over the entire layer thickness. Interaction can result in an increased average thickness of the layer and a change in refractive index value. In practice both these effects are taken into account simultaneously when approaching the effective refractive index change. However, as schematically shown in Fig. 1.1(a), a considerable amount of the evanescent field extends outside the sensing layer, making the N_{eff} value sensitive to the refractive index of the solution above the sensing layer.

Figure 1.1: Schematic representation of evanescent field sensing (a) surface sensing and (b) homogenous sensing (after Ref. [1]).

Surface sensing has found application as planar evanescent waveguide sensors mostly used for the detection of ultra-thin biological molecular layers [13], [14] of thickness smaller than the wavelength (λ) in vacuum immobilized on the surface of a waveguide. Such sensing scheme is currently the subject of interest in chemical sensing where the optical-chemical transducing mechanism involves an ultra-thin surface layer. [15] The sensitivity in such configuration is related to the squared field magnitude at the waveguide-cover interface. [16] Optimizing the sensor sensitivity requires a suitable choice of the waveguide, substrate refractive index, and

the waveguide thickness relative to the wavelength, which in turn maximizes the squared modal field at the surface. [17]

In contrast, the diagram of Fig. 1.1(b) shows a thick sensitive layer such that the power of the evanescent field outside the sensitive layer can be neglected, leading to a homogenous sensing. Evanescent wave sensing of a chemical or physical quantity which is homogeneously distributed in the semi-infinite waveguide cover refers to a different electromagnetic condition. The sensitivity is now related to the integral of the squared evanescent field in the cover material. [16] This type of sensor is insensitive to the refractive index of the solution due to the evanescent field being entirely confined inside the sensing layer. Such waveguide refractometric sensors can be used in gas or liquid concentration monitoring, for measuring traces of chemicals by means of a thick selective membrane [18] and, more generally, for measuring all physical/chemical quantities whose variation corresponds to a change of refractive index.

1.4.2 Sensing layer structure

A comparison between two sensing methods of the previous section reveals a potential advantage of the homogenous sensing: any change in sensing layer thickness will strongly affect the overlapping power integral of the evanescent field in the cover region. Unfortunately the application of standard thin films as a sensing layer does not allow one to take advantage of homogenous sensing. By increasing the sensing layer thickness, the evanescent field propagation losses due to scattering and absorption will increase; and also, a homogenous concentration or distribution of the measurand into the sensing layer would become impossible. Thus, finding a thin film structure able to overcome these limitations becomes

a challenging task in both aspects of optical waveguide design and fabrication-technology. [3]

A possible solution to this problem would be the deposition of some specially structured dielectric materials with a strictly controlled refractive index, porosity, structural configuration and high surface area to volume ratio. From the existing technologies, such films can be deposited using the GLAD technique. As shown in Ref. [4], this technique offers a high degree of control over the film microstructure. Porosity is created in these films by enhanced self-shadowing during the growth process where a random growth fluctuation in one area produces a shadowed region where subsequent incident flux cannot reach and a high point where incident flux may deposit, creating preferred growth. By producing a symmetrical and highly oblique incident flux, shadowing can be greatly enhanced and highly porous films can be fabricated with vertical columns and porosities much greater than those fabricated with a one sided flux. [19]

1.5 Nanostructured thin films in optics

Optical properties of films deposited at oblique as well as glancing angles have been an object of research for more than five decades. Experiments in the early stages [20], [21] were inspired by the idea that optical properties of these films can be easily modified by changing deposition conditions and hence the film structure itself. Thin films deposited at an oblique angle onto stationary or rotating substrates were found to display optical activity with anisotropy and direction-dependent absorption. Later on, the discovery and characterization of birefringent properties of transparent metal oxides [22], [23] deposited at oblique angles was an important aspect in the application oriented development of such films. Films of TiO_2 , ZrO_2 [24] and Al_2O_3 demonstrated higher preferential absorption of one

polarization over another, thus opening up an entire new era for further development and applications. In addition, the use of the GLAD technique to deposit optical films with the ability to produce well-controlled columnar nanostructures, resulting in artificially engineered optical materials, enhanced the potential even further. The unique properties of structured materials lead to a wide range of potential applications.

1.5.1 Optical properties and applications

From the range of physical and optical properties that structured thin films have demonstrated, perhaps the most straight forward applications come from the enhanced surface area inherent to these films. The columnar microstructure of such films increases the surface area as compared to a dense film. A variety of methods have attempted to measure the surface area of structured films, with results showing a very strong dependence of surface area on the flux incidence angle. To demonstrate, a 925 nm thick silicon dioxide (SiO_2) GLAD film with slanted columns deposited at the incidence angle $\alpha=85^\circ$ resulted in an approximate surface area of 119.5 cm^2 for 1 cm^2 substrate. [25] In another application, high surface area ($\sim 1000 \text{ m}^2/\text{g}$) [26] in carbon-based capacitors were reported, providing capacitances in the range of 100-200 F/g. However, as pointed out in Ref. [27], the surface area enhancement is not significantly influenced by the particular structure fabricated, but rather is linked to the deposition angle.

The enhanced surface area observed in GLAD films has been exploited in a variety of applications such as catalytic surfaces in the automotive industry, [27] fluid concentration and humidity sensing, [28] super capacitors, [26] solar cells [29] and thermal barrier coatings. [30] In particular, one promising application of GLAD films is in fabricating sensor devices. Due to their inherently porous nature,

gaseous species in the ambient environment are free to diffuse through the structure producing detectable changes in the dielectric constant of the system. The surfaces of oxide films such as TiO_2 and Al_2O_3 fabricated using GLAD are intrinsically hydrophilic, promoting the capillary condensation of water vapor within the porous structure. [31] Because of the large pore sizes in the GLAD structures, diffusion is not inhibited and the vapor can rapidly adsorb onto or desorb off of the surface, [32] demonstrating moisture adsorption in biaxial and chiral optical thin film coatings. This led to response times which are significantly faster than in conventional devices [33] with a desorption response time of 42 ms.

In addition to enhanced surface area, the optical anisotropy of these films is probably the most exploited property which led to many optical device applications. By manipulating the porosity of the film along the substrate normal, the planar density of a film can be tailored, making this technology capable of generating interference coatings of different refractive indices using a single source material. Therefore, it presents an alternative to the common method of creating optical interference coatings with specific transmission and reflection properties by depositing multiple layers of isotropic materials with different refractive indices. [34] To demonstrate, the GLAD technique was used to fabricate optical filters of different configurations such as chiral polarization [35] and sinusoidal index gradient, also known as rugate filters. [36], [37] The periodic refractive index of these filters leads to a Bragg reflectance and an optical stop band. Also, the porous nature of these coatings makes the device sensitive to the voids environment, leading to another sensing application as demonstrated in a high speed optical humidity sensor. [28]

The ability to control the orientation of the columns of the film means that the direction of the anisotropy can be tailored at every point in the film. This

property has been exploited, resulting in films that demonstrate optical activity in rotating linearly polarized light as well as creating circular Bragg phenomena. For example, helical films exhibit selective reflection of circularly polarized light and optical rotation of linearly polarized light. When the columns are sculpted into helical columnar shapes, the planar anisotropy rotates with thickness in a similar manner, as is seen in chiral nematic phases of liquid crystals. [38], [39] This periodicity distinguishes between left and right circularly polarized lights. [40] Based on this phenomena, polarization filters and mirrors, [41] photonic bandgap crystals, [42] as well as sensing devices [43] where fluids penetrating the void region affect the optical response of the film, have been demonstrated.

Antireflection coatings are another important application of optical thin films. Although antireflection coatings are widely available, in most cases they function only at a single wavelength and only when the light source is perpendicular to the film surface. One approach to fabricating these coatings is with a graded-index layer that presents a smooth transition from the refractive index of the incident medium to the substrate index. [44], [45] Using the GLAD technique, graded-index broadband omnidirectional coating was created by depositing silica nanorods. In another application, [46] SiO_2 films with a Gaussian density and refractive index profile were deposited onto glass substrates. The smooth index transition introduced by these films virtually eliminates the Fresnel reflection losses which would otherwise occur at the air-glass substrate.

1.5.2 Nanostructured film as cladding layer

As evident from the previous section, obliquely deposited thin films behave as an uniaxial-appearing layer to a normally incident light, showing undisputable optical birefringence properties. However, the review of many works led to a

surprising observation: a normally incident light propagating into the film is one common aspect of all studies, thus leaving untouched the material behavior and characterization in cases when light propagates parallel to the film surface. To the best of the author's knowledge, there is a lack of published works to undertake this issue, resulting in an unaddressed potential of such applications.

In part, it is the purpose of this work to develop applications where the evanescent part of the optical field propagates into the nanostructured film parallel to the substrate surface. The porous nature of sensing layer makes the device sensitive to the voids environment; as it propagates along the film, the evanescent field drawn deep into the porous layer probes the environment. The ability to control the film refractive index and structure can provide enhanced interaction of the optical field with the sensing environment, benefiting from optical properties inherent to GLAD films.

1.6 MZI for optical detection

Once the sensing element of the sensor captures the target material, a wide variety of signal transducing methods can be applied to detect and measure its presence. These methods can generally be classified as optical, electrical, or mass-based measurements. Amongst the variety of optical methods, those that produce a direct signal affected by the molecular interaction have become increasingly popular in academia and industry. [11] On the basis of these methods is the measure of refractive index change when a captured object binds to a surface-attached receptor or simply fills (replaces) part of the original material in the sensing layer, without the use of any type of labeling. To demonstrate, a few of the many techniques that fall into the optical transducing category are based on: (a) interferometry, with Mach-Zehnder, Young's, [47] and Michelson interferometers; [48]

(b) mode coupling, with fiber Bragg grating; [49] and (c) the resonance, with Fabry-Perot, [50], [51] photonic ring, [52] and surface plasmon resonators. [14]

From the group of optical approaches, the systems based on interferometry have attracted a wide interest, with the MZI being the most exploited one. Although very good sensitivities are achieved with Bragg grating and surface plasmon resonator devices, the interferometric methods have shown the highest sensitivity potential. [53], [54] Based on this method, many different types of IOS have been implemented and successfully applied for different applications of chemical sensing. They perform reasonably well and its basic structure with chemo-optical interface layer is relatively simple to implement in integrated optics. High resolution of the refractive index measurement in the range of $\Delta n \approx 7 \times 10^{-6}$ and very low detection limits have been achieved. [55] It also can provide an internal reference for compensation of several perturbing factors causing refractive index fluctuations, such as temperature variations and laser power output fluctuations. In addition, the MZI structure has found many other applications [56] such as modulators, optical filters and switches in planar lightwave circuits.

The basic structure of a MZI is schematically shown in Fig. 1.2. The input light is coupled into a linear waveguide where it propagates until reaching a Y-splitter. At this point the signal splits following two different paths which are shown as the MZI arms. At the second Y-junction, signals from two arms are recombined, with the resulting signal depending on the optical path length difference. The output power (P_{out}) at the MZI output depends on the phase difference ($\Delta\phi$) of both signals at the junction area. As a result, a constructive or destructive interference will produce sinusoidal variation in the output intensity.

The advantage of the MZI in various applications is based on the ability to control the optical path difference between arms by changing the refractive index

in one of them. Any refractive index change in one arm will strongly affect the output intensity. By locally removing the cladding layer in the sensing region, an interaction between the evanescent field of propagating mode and the material to be sensed can be achieved. Large interaction length values will of course result in enhanced sensitivity.

Figure 1.2: Schematic of the basic configuration of a MZI with structured thin film (STF) deposited in the sensing window.

The principle of operation of a MZI can be found elsewhere, [57] however a brief overview is presented here. Assuming the first Y-junction acts as an ideal splitter and splits the input signal A_0 into two equal parts, then the two signals propagate along different paths, with an optical phase difference $\Delta\phi$ introduced in the lower arm of the MZI. Therefore, signals before the second Y-junction will have the form:

$$A_1 = A_0 e^{j\omega t} \quad (1.1)$$

$$A_2 = A_0 e^{j(\omega t + \Delta\phi)} \quad (1.2)$$

where $\omega = 2\pi f$ is the angular frequency of optical field oscillation. After the interference at the second Y-splitter, the output signal can be expressed as:

$$A_{out} = A_1 + A_2 = A_0 (e^{j\omega t} + e^{j(\omega t + \Delta\phi)}) \quad (1.3)$$

As Eq. (1.3) shows, the output signal depends on the phase difference between two arms. After derivation, [58] power at the output can be expressed as:

$$P_{out} = \sum P_{in,k}(1 + \cos \Delta\phi_k) \quad (1.4)$$

where $P_{in,k}$ is the input power of the k -th mode and the $\Delta\phi_k$ is the measurand induced phase difference related to mode k .

For the case where only one mode is propagating, the transfer function of the MZI, or the power ratio (P_{out}/P_{in}), can be seen as a sinusoidal function of $\Delta\phi$, with the quadrature points occurring when the phase difference between the two arms of the interferometer is $\Delta\phi = (n + 1/2)\pi$, for $n=0,1,2,\dots$. Note that the quadrature points correspond to the maximum slope or variation in output signal.

For a given transfer function of a MZI, the effective refractive index change ΔN_{eff} induced by the refractive index change within the evanescent field region can be obtained with:

$$\Delta\phi = \frac{2\pi}{\lambda_0} L_{int} \Delta N_{eff} \quad (1.5)$$

where λ_0 is the wavelength of light in free space and L_{int} is the interaction length as shown in Fig. 1.2. With this relation, the actual ΔN_{eff} can be estimated by measuring the change in the fringe period of MZI transfer function. Similarly, Eq. (1.5) can be used to approximate the actual path length difference in between arms of an unbalanced MZI.

The sensor response scales with the squared amplitude of the electric field within the perturbation area, [16] therefore just a small fraction of the total modal power in the near surface volume where the dielectric constant has been modified will affect the phase of the output signal. However, an increase in power fraction can be achieved by increasing the modal interaction length, or the interaction

surface, thus providing a significant cumulative effect.

The device proposed in this project explores an approach where an increase in the evanescent field interaction with the measurand is achieved with the application of a dielectric structured thin film. A large surface area and the excellent optical properties of these films provide an increase in the interaction surface as well as a better environment which pulls the evanescent field deep into the perturbation volume, thus potentially increasing the evanescent field sensitivity of a MZI sensor.

Chapter 2

Technology and Material Considerations for IO Sensing

Due to their platform and design characteristics, IOS devices share the same technology and materials applicable to IC fabrication and planar waveguides. From the range of planar waveguides available, the silicon nitride with its high core refractive index and a relatively simple fabrication process has recently attracted attention especially for sensing applications. [55]

In this section, a short discussion of Si_3N_4 waveguide is presented, with the focus on optical sensing and device sensitivity. Core-cladding refractive index difference is of great importance when it comes to optical sensitivity. After a careful consideration, three dielectric materials, aluminum oxide, zirconium oxide, and titanium oxide were selected as cladding layer materials. In addition to their good optical characteristics, the application of the GLAD technique on the fabrication process makes these oxides suitable for sensing layer.

2.1 Silicon nitride planar waveguides

Planar waveguides with a Si_3N_4 core surrounded by SiO_2 cladding layers represent one of the best alternatives to fabricate waveguides with large core-cladding refractive index difference. This technology employs silicon substrates but makes use of Si_3N_4 for forming the core layer of the waveguide. [59] The refractive index of Si_3N_4 layer can be anywhere in the range of 1.90 to 2.01 depending on the relative concentration of hydrogen and nitrogen atoms within the deposited material. This flexibility allows for fabrication of waveguides with large core-cladding refractive index difference $\Delta = (n_{12} - n_{22})/n_1$ without adding extra loss. It also makes them attractive for sensing applications where a number of dielectric materials with their relatively high refractive index would qualify as sensing layer.

Low scattering and absorption losses and compatibility with silicon processing makes this technology advantageous for integrated optics circuits. [56] Another advantage of forming Si_3N_4 waveguides on SiO_2 buffer and silicon substrate is that the well-developed silicon technology can be employed. Also, the possibility of integrating these optical devices with microelectronic and or micro-mechanical elements is another advantage.

The Si_3N_4 thin films can be deposited using a plasma-enhanced chemical vapor deposition (PECVD) process with silane (SiH_4) gas as the base material and gases such as nitrous oxide (N_2O) and ammonia (NH_3). The main design considerations are waveguide uniformity and low propagation losses. This technology is advanced enough to provide very good waveguide uniformity with losses depending on core-cladding refractive index difference. Propagation losses are typically below 0.2 dB/cm but increase considerably in the spectral region near $1.5 \mu\text{m}$ because of the absorption caused by vibrational overtones of the N-H and Si-H bonds.

Other technologies such as silicon on insulator (SOI) [60], [61] and silica on

silicon [62] were also considered; however, their core refractive index is either too high (SOI), or too low (silica on silicon) when it comes to matching with the refractive index of dielectric materials in sensing layer, thus compromising the overall device sensitivity.

It is generally assumed that the sensitivity of a guided wave to changes of the cladding index is smaller or much smaller than that of a free-space beam. [17] This is based on the fact that only a small fraction of the optical field guided in the waveguide propagates in the sensing medium. In this context, the modal sensitivity can be defined as the ratio of the effective index change of the guided mode to the refractive index change of the cladding layer. Using a mode solver 2D from OptiBPM [63] simulator, the guided mode effective index of a Si_3N_4 rib waveguide with refractive index $n=1.92$ and thickness $t=1\ \mu\text{m}$, and SiO_2 buffer of $n=1.46$ and $t=3.5\ \mu\text{m}$ was simulated for a range of cladding layer refractive index (n_{SL}) values, with results plotted in the double axis graphs of Fig. 2.1.

The slope (S) given with:

$$S = \frac{\Delta N_{eff}}{\Delta n_{SL}} \quad (2.1)$$

presents the device sensitivity. As the graph shows, higher sensitivities can be achieved when cladding-core refractive indices get closer; of course, it becomes easier to pull the evanescent field outside the core layer deep into the cladding. Very little change in the cladding refractive index would strongly affect the field distribution inside the waveguide. This is also demonstrated in Fig. 2.2, where each plot shows the fundamental mode corresponding to a refractive index value in the cladding layer. By reducing the core-cladding refractive index difference, the fundamental mode extends deeper into the cladding.

As demonstrated from these graphs, one of the advantages in using a Si_3N_4 waveguide for sensing applications originates from its refractive index and

Figure 2.1: Waveguide effective refractive index as function of cladding refractive index.

Figure 2.2: Electric field in a silicon nitride waveguide for different values of cladding refractive index, ranging from 1.6 to 1.86.

the possibility of matching it with the refractive index of a broad range of dielectric oxides. From this perspective, an ideal cladding layer should have a refractive index close to the core one, and at the same time it should be porous enough to enhance the evanescent field-measurand interaction, thus maximizing the effective refractive index change of the evanescent field propagation path. As will be demonstrated in the following, a dielectric structured thin film fabricated with the GLAD technology is a good candidate for optical sensing applications, presenting a combination of sensing principles with detection techniques where the technology can make a difference.

2.2 Structured thin film fabricated with GLAD technology

Most thin film applications in the microelectronics or optical industries require dense and durable coatings for optimum performance. However, for some applications such as thin film catalysts or sensors, a structured thin film would be desirable. For example, tin dioxide, aluminium oxide, [64] and silicon [65] GLAD films have been used in a number of gas or liquid sensor applications. A common desired characteristic of these porous films is a high ratio of surface area to volume, leading to devices with optical properties very sensitive to a change in the content of the pores. A very large specific surface [40] of such porous devices, compared to conventional bulk dielectric thin film structures, suggests their applications in chemical and possibly biological sensing.

Typically, there are two group methods of creating highly porous films: by

etching a dense material using methods such as galvanostatic, chemical, or photochemical, and by directly depositing (growing) a structured porous film on the substrate surface. The synthesis of a thin film by chemical etching generally involves two processing steps: dense film deposition, and chemical etching. Generally, two different techniques such as chemical vapor deposition (CVD) or physical vapor deposition (PVD) can be employed in the deposition process. Further, depending on the film material and its application, there are different etching techniques able to produce porous films with very well defined thickness. As to demonstrate, porous anodic alumina films with hexagonal pore ordering were realized through a two-step anodization process. [66] In other examples, porous silicon was synthesized using chemical etching in the presence of hydrofluoric acid (HF). [67] However, while this is a very wide area, the purpose of these remarks is to briefly mention it as an existing alternative to GLAD technique, which is the technique of choice for this work.

The GLAD process combines PVD at highly oblique vapor incidence angles with controlled substrate motion, enabling the growth of various complex nanostructures such as helices, square spirals, chevrons, and vertical posts. [19] Porosity in these films is created by enhanced self-shadowing during the growth process where a random growth fluctuation in one area produces a shadowed region where subsequent incident one-sided flux cannot reach and a high point where it may deposit, thus creating preferred growth. In general, there are some advantages of GLAD versus anodic techniques: higher surface ratio per volume, more options and controllability over the film microstructure, and better control on film refractive index.

In regard to the GLAD deposition technique, depending on the relative position between source material and the substrate, two methods can be distinguished:

(a) a film deposited onto a stationary oblique substrate, with the resulting morphology of columns with an angle depending on the substrate obliquity; and (b) a film deposited onto a rotating substrate oriented obliquely with respect to the flux of incoming vapor (as depicted in Fig. 2.3). It has been observed that films produced with a highly oblique incident flux from one side have a porous microstructure, [68] but the limited porosity and the inclined orientation of the film microstructure have restricted their usefulness. [69] On the contrary, by producing a symmetrical and highly oblique incident flux, shadowing can be greatly enhanced and highly porous films can be fabricated with vertical columns and porosities much greater than those fabricated with a one sided flux.

Figure 2.3: Schematic representation of GLAD process (a) relative position of substrate and vapor flux from source material, and (b) film deposited onto the substrate with columns and shadowed regions where the position of individual columns is determined by the randomness of the nucleation process.

However, for the work presented here, GLAD films were deposited onto both stationary and rotating substrates. While deposition on stationary substrates served as a starting point and a source for preliminary data, deposition on rotating substrates was employed in several prototypes when structured thin films with tilted and vertical columns were created.

2.3 Dielectric materials for structured thin films

As pointed out in Section 2.1, a small core-sensing layer refractive index difference would achieve higher optical sensitivities. It is for that reason that choosing the right dielectric materials would be essential for this application. From the range of dielectric materials available, three of them were considered: aluminum oxide, zirconium oxide, and titanium oxide. Due to their good optical properties, these materials have found applications for laser manufacturing and optical coatings. A brief overview of optical properties and the refractive index range of these materials are shown.

Aluminum oxide is a medium refractive index (Fig. 2.4) and low absorption material suitable for coatings in the near-UV to IR regions. Typical applications include near-UV laser anti-reflection and dielectric mirror designs. Completely oxidized alumina films are absorption-free in the range of 0.3-5 μm . Slight dissociation and oxygen loss occurs during evaporation. Adhesion is good to glass, most other oxides, and some polymers. The films generally grow with a crystalline microstructure and low packing density and exhibit index changes when vented to moist air. More on physical and optical properties of Al_2O_3 can be found at Cerac Inc. documentation which was the supplier for Al_2O_3 and other dielectrics as well.

Zirconium dioxide has a relatively high refractive index (Fig. 2.4) and low absorption; it is widely used for coatings in the near-UV to IR regions. Dense layers with exceptional hardness can be deposited by electron-beam evaporation. Films of ZrO_2 can be used in combination with silicon dioxide layers to form high index-contrast multilayered structures. Completely oxidized ZrO_2 films are absorption-free over the range 0.3-8 μm . Adhesion is excellent to glass and most other oxides. The films generally grow with a crystalline microstructure. Under

Figure 2.4: Refractive index of Al_2O_3 , ZrO_2 , and TiO_2 thin films as function of wavelength

some evaporation conditions, such as low energy resistance-heated evaporation, low substrate temperature, or excessive background pressure, the films grow with sufficient void volume (packing density < 0.8) to exhibit index changes when vented to moist air. In general, the refractive indices are dependent on the degree of oxidation and the film density achieved.

Titanium dioxide has two particularly desirable qualities: it provides the highest index film material for the visible and NIR region (Fig. 2.4), and also is hard and stable in combination with other oxides including SiO_2 films where tensile stress of TiO_2 layers is balanced by the compressive stress of the silica layers. As a result, this material has gained extensive interest for its many applications, such as catalyst, gas sensor, [70] and optical and optoelectronic devices. TiO_2 thin films are especially useful for optical devices due to their desirable properties such as a low absorption coefficient and a high transparency in the visible and near-IR

region. [71]

2.4 Summary

In this Chapter, the waveguide technology, the technique for sensitive layer deposition, as well as the appropriate dielectric materials for optical sensing applications were outlined. The refractive index offered by Si_3N_4 , with its ability to adjust to different applications, and the compatibility of Si_3N_4 waveguide fabrication with IC technology, made this the technology of choice. Further, deposition of a dielectric porous film using GLAD technique can provide sensing layers with a very large ratio of surface area to volume, able to enhance the interaction of a propagating evanescent field with the measurand. The dielectric materials selected for this application offer excellent optical properties with low absorption and scattering losses, good adhesion to Si_3N_4 and other oxides, and a broad range of refractive indices.

Chapter 3

Device Modeling and Simulation

Modeling and design of an interferometric-based evanescent field sensor is outlined in this Chapter. In addition, other conceptual devices were explored. [5] The application of a novel optical field sensitivity mapping technique enabled first order optimization of device geometrical parameters with the final goal of enhancing the overall optical sensitivity. The technique is simple, requiring only two simulations of optical field propagation and the extraction of a sensitivity map. The method was demonstrated in a design optimization process of a realistic multi mode interference sensing device. A comparison between the optimized and two other devices of different length showed a 10 dB higher dynamic range of the output power ratio characteristic and better linearity, demonstrating enhanced sensitivity of the optimized device.

Although the conceptual design explored in Ref. [5] demonstrated good optical sensitivity with a high dynamic range, a more conservative approach was considered for this project. Interferometric sensing with a MZI has a proven history, thus it can serve as a vehicle to achieve the goal of this project, to validate a newly proposed optical sensing platform. The design process starts with modeling and simulation of a single mode Si_3N_4 rib waveguide with a given set of refractive

indices. The waveguide profile and dimensions obtained in Section 3.1 are the basis for the design of optical sensing devices such as segmented waveguide and MZI.

The dielectric structured thin film growth is simulated in Section 3.2. Microstructural growth orientation, film density and refractive index, as well as film anisotropy are evaluated. These parameters provide settings for further simulation of MZI device (Section 3.3) with structured thin film deposited on the sensing area, and also for thin film deposition and fabrication. Finally, simulation results are summarized to draw some guidelines for the next step of device fabrication.

Note that throughout the chapter, modeling and simulation of waveguide profile, linear segmented waveguide and MZI is completed with OptiBPM [63] software. Furthermore, modeling of structured thin film growth based on the GLAD technique is simulated with 3D-FILM [72] software.

3.1 Waveguide profile

During the course of this work, a feasibility discussion led to the decision that an existing mask with straight waveguides and MZIs will be used, instead of designing and fabricating a new one. Therefore, several designing parameters such as the width of the waveguide ($w=5\text{ }\mu\text{m}$), Y-junction geometry, and the length of MZI arms were predefined, leaving the rest of the parameters to play with. In addition, starting silicon substrates with a $3.5\text{ }\mu\text{m}$ thermal oxide layer ($n=1.46$) were made available and are considered throughout the simulation. Other refractive index values considered are $n=1.92$ for Si_3N_4 core and $n=1.45$ for SiO_2 cladding. Unless otherwise stated, simulations are completed for Gaussian input plane at wavelength $\lambda=1.62\text{ }\mu\text{m}$.

Interferometer-based evanescent field sensors typically require single-mode operation. If several modes are propagating through the waveguide of the device, a change in the measurand content in the active region would affect the phase of all modes by causing intermodal coupling, thus the intensity of the output signal can no longer be considered as indication of the refractive index of the measurand.

3.1.1 Modes of a rectangular waveguide

Most practical applications require confinement of light in two dimensions, such that an optical signal can be routed along a specified path. The additional horizontal confinement can be achieved using a rib waveguide structure. It is important to note that even though pure transmission electric (TE) or transmission magnetic (TM) modes do not exist in a rectangular waveguide, as is the case with a slab waveguide, one can divide the modes into two sets of hybrid modes: quasi-TE modes, for which all field components are assumed to exist except for E_z , and quasi-TM modes, for which all components are assumed to exist except for H_z . An analytical solution to the two-dimensional wave equations of a rib waveguide is generally not possible due to the complexity of the boundary conditions. However, several techniques for numerical and semi-analytical solutions have been studied.

The simplest semi-analytical approach is the effective index method. [73] This method makes use of the typical practical situation in which the thickness of a rectangular waveguide is much smaller than its width ($d \ll w$). The single-mode condition can be provided with the normalized parameter V [56] given by:

$$V = k_0 \sqrt{N_1^2 - N_2^2} < \frac{\pi}{2} \quad (3.1)$$

where V is the normalized frequency, k_0 is the free-space wave number defined as

$$k_0 = \frac{\omega}{c} = \frac{2\pi}{\lambda}, \quad (3.2)$$

λ is the vacuum wavelength, and N_1 and N_2 are effective index values of core and cladding regions. For a rib depth of 110 nm, the mode solver 2D gave $N_1=1.8314$ and $N_2=1.8251$. With these values, Eq. (3.1) gives a normalized frequency $V=1.47$, which is below $\pi/2$ value.

Although Eq. (3.1) is referred as the single-mode condition for a rectangular waveguide, it is important to note that it is an approximated calculation, and should not be considered sufficient in providing single-mode operation of the waveguide. Instead, numerical simulations using mode solver 3D were completed for the rectangular waveguide structure.

The Mode Solver 3D program found the effective refractive index and the modal field of the fundamental guided mode in a waveguide with profile shown in Fig. 3.1, indicating that the structure does not support any higher order modes. The input field was Gaussian with half width $(x,y)=(5,0.5)$ and center $(x,y)=(0,0.5)$.

Figure 3.1: WG profile simulated with mode solver 3D with rib thickness 110 nm and refractive indices: SiO_2 buffer $n=1.46$, Si_3N_4 core $n=1.92$, and SiO_2 cladding $n=1.45$.

The solution was obtained with a correlation function method; [74] it calculates, during propagation of a starting field determined by the user, the correlation integral between the starting field and the propagating field at every point, thereby creating the field amplitude correlation function for the waveguide. The method provides all the information required for a complete modal description of the fields, including propagation constants, weights of each mode, and the mode eigenfunctions. As a result, mode index $N=1.8337$, confinement factor of 82%, and *weights*=1 were found. Simulation results for fundamental TE and TM modes are shown in Fig. 3.2.

The outcome of this simulations would be a waveguide with profile schematically shown in Fig. 3.3. It consists on a $3.5\ \mu\text{m}$ thick thermal SiO_2 layer, a $1\ \mu\text{m}$ Si_3N_4 layer (with 110 nm etched away to create the waveguide rib), and a $1.8\ \mu\text{m}$ thick SiO_2 cladding. Note, the $5\ \mu\text{m}$ rib width was extracted from the mask layout. Unless otherwise stated, this waveguide profile would be used in the following devices.

3.2 Structured thin film growth model

Historically, computer simulation of thin film growth has been limited by insufficient computing power, binding the simulators to simple two-dimensional [75], [76] and three-dimensional [77], [78] models which did not take into account the effects of surface diffusion of adatoms over large scales. With the improvements in computing power this limitation has been lifted, allowing the development of programs capable to model structured thin films created at high flux incidence angle. Although a number of software packages have recently been developed, [79], [80] one simulator that has proven quite successful in the modeling-simulation of films

(a)

(b)

Figure 3.2: Intensity distribution of the fundamental modes of the waveguide structure (a) TE field, and (b) TM field.

Figure 3.3: Schematic representation of waveguide structure with refractive indices: $n=1.46$ for SiO₂ under cladding, $n=1.92$ for Si₃N₄, and $n=1.45$ for SiO₂ over cladding.

deposited at glancing angles is the Monte Carlo ballistic simulator program 3D-FILM; [72] it evolved from a two-dimensional simulation package called SIMBAD.

The 3D-FILM simulates micro structural growths with the capability of modeling faceted grain growth due to anisotropic adatom mobilities and surface energies. It represents the film as an aggregation of large blocks, with each block presenting the average behavior of approximately 1000 atoms. Particles are serially launched from just above the growing film and follow straight line trajectories until they strike the substrate or the film. The particles aggregate into the film after a short surface diffusion to minimize a surface curvature-dependent chemical potential. The combination of ballistic shadowing and short range surface diffusion successfully accounts for the formation of the columnar thin film microstructure characteristic of refractory materials at typical deposition temperatures. Simulation of such models produce a three dimensionally grown film which can be displayed graphically as 3D images.

Simulation starts with the declaration of substrate configuration and parameters controlling the deposition process. Substrate is seen as simple cubes able to create a variety of configurations such as flat substrate, trenches, ridges, and steps necessary to model an optical waveguide. The process continues with the film growth, where large amount of details are generated, typically consisting of millions of cubes. The simulator creates viewing information in the form of simple 3D objects such as triangles and quadrilaterals, each having defined properties as color and transparency.

The structured thin film growth at incidence angle 60° , 70° , and 85° was simulated in a surface area $10 \times 10 \mu\text{m}^2$ with results shown in Figs. 3.4 and 3.5. The typical diameter of columns was found to be approximately 100 nm. A simple geometrical approach expressed with Eq. (3.3) showed that the flux angle of incidence with the substrate normal (α) and the predicted angle of inclination (β) of column growth could be related for evaporated thin film growth at high incidence angles ($\alpha > 60^\circ$) and limited surface diffusion. [81]

$$\beta = \alpha - \sin^{-1}\left(\frac{1 - \cos \alpha}{2}\right) \quad (3.3)$$

The expression proposed in Eq. (3.3) is referred as Tait's rule; it assumes deposition on hemispherical topped columns with the center of the column portion receiving the flux, thus determining the direction of growth. This argument was based on observations of ballistic deposition models, which were found to closely resemble real film behavior. This equation has been shown to predict $\beta(\alpha)$ relation well for Fe and MgF_2 films, and correctly reproduced the general observation that β approaches a value less than 90° as $\alpha \rightarrow 90^\circ$. [82]

The implementation of Tait's rule in 3D-FILM to obtain column inclination angle is relevant to this work. Based on this, we were able to model the relation of

Figure 3.4: Simulation of structured thin film deposited at incidence angle $\alpha=60^\circ$ viewed from (a) above, and (b) side.

flux incidence angle of deposition with the film density. After a growth process is completed, film density and surface area corresponding to a sampling volume was determined. Following this procedure, density in a sampled volume of dimensions $10 \times 10 \times 1.5 \mu\text{m}^3$ was evaluated. Density values of 75%, 65%, and 34% of bulk material, corresponding to incidence angles 60° , 70° , and 85° were obtained. Note that the percentage value expresses the content of bulk material in the film volume with the rest being void (air).

(a)

(b)

Figure 3.5: Models of structured thin film deposited at incidence angle (a) $\alpha=70^\circ$, and (b) $\alpha=85^\circ$.

Knowing the percentage volume of bulk material and void, the Bruggemann effective medium approximation [83], [84] can be applied, allowing determination

of the refractive index of the heterogeneous mixture of two components. In its general form, Bruggemann's approximation would be:

$$v_d \frac{n_d^2 - n_x^2}{n_d^2 + 2n_x^2} + v_p \frac{n_p^2 - n_x^2}{n_p^2 + 2n_x^2} = 0 \quad (3.4)$$

where v_d and v_p are volume fractions of deposited material and pore contents, with $v_d + v_p = 1$; the n_d and n_p are the corresponding refractive indices for pure bulk deposited material and pore content, and n_x is the porous film index.

By solving Eq. (3.4) for n_x , it was possible to evaluate the refractive index of simulated films for given volume fraction and material refractive index of deposited material. Calculated refractive index values for structured thin films of three dielectric materials and three different deposition angles are summarized in Table 3.1. Note that the refractive index values in the second column correspond to the bulk material (deposited at $\alpha=0^\circ$) at wavelength $\lambda=1.6 \mu\text{m}$, and are extracted from data of Fig. 2.4.

α/v_d	$0^\circ/1$	$60^\circ/0.75$	$70^\circ/0.65$	$85^\circ/0.34$
Al_2O_3	1.61	1.56	1.51	1.29
ZrO_2	1.95	1.83	1.71	1.44
TiO_2	2.25	2.06	1.89	1.56

Table 3.1: Refractive index values of structured thin films calculated with 3D-FILM and Bruggemann's approximation.

From these results, it is evident that film refractive index can be tailored to a desired value by adjusting the incidence angle in the deposition process. This would allow a better match for core-cladding refractive index difference, and also for adjusting the anisotropic properties of the layer.

While simulation of films in Figs. 3.4 and 3.5 was completed for deposition on a stationary substrate, 3D-FILM can model deposition on rotating substrates as

well. A combination of at least two model parameters such as angle of incident flux and substrate rotation leads to different film structures. For instance, films shown in Fig. 3.6 were created by rotating the substrate at two different velocities, resulting in films with a variety of structures, including one with vertical columns which is of interest and is considered during the fabrication process.

Figure 3.6: Structured thin films deposited onto rotating substrate at different velocities producing (a) vertical columns, and (b) spirals.

3.3 MZI for optical sensing

Simulation of the MZI for optical sensing was made possible with the application of the effective index method. [73], [85] It is based on the effective refractive index and the modal field of the fundamental guided mode generated for ridge and cladding sections of a waveguide profile with layer structure as described in Section 3.1. The layout and the refractive index profile (on xz -plane) of a typical MZI model is shown in Fig. 3.7, with dimensions extracted from the mask layout. While arms have identical geometrical lengths, there is a difference in the optical paths. This difference is induced by a different cladding material (and refractive

Figure 3.7: Refractive index topology in the MZI with symmetrical arms and sensing window of length $L=3.4$ mm. Note: dimensions not to scale.

Material	n	WG N_{eff}
N ₂	1.000292	1.85157304
CO ₂	1.000449	1.85159257
Difference (Δ)	$1.57e - 4$	$1.95e - 5$

Table 3.2: Δn and ΔN_{eff} when N₂ in sensing layer is replaced by CO₂.

index) in a section of length $L=3.4$ mm on the lower arm. This section models a sensing region where SiO₂ cladding has been removed and replaced by a structured thin film layer deposited with the GLAD technology. Any change in the refractive index of the material filling film voids will result in effective index change of the entire section.

The effective refractive index values obtained in Section 3.1 were set for cladding ($N_c=1.8314$), and ridge ($N_r=1.8337$) sections. In sensing region, a TiO₂ structured thin film of thickness $1.4 \mu\text{m}$ and density 34% ($n=1.56$) was assumed. The refractive index values at $T=0^\circ\text{C}$ and atmospheric pressure for nitrogen (N₂) and carbon dioxide (CO₂) introduced into the sensing layer as well as the corresponding effective refractive indices of waveguide stack are summarized in Table 3.2.

With these effective refractive indices and a sensing area of length $L=3.4$ mm,

the MZI model was simulated. The procedure involved two consecutive runs, each for N_2 and CO_2 gas in the sensing layer. The wavelength dependent intensity obtained for each gas is shown in Fig. 3.8, where a phase shift of $\Delta\phi=10$ nm is induced as a result of the effective refractive index change in the sensing region. The change of gas content into the sensing environment is reflected at the output intensity by shifting the wavelength response of the MZI.

Figure 3.8: Wavelength dependent output intensity

Furthermore, device sensitivity was modeled by simulating the output intensity as a function of effective refractive index change in the active region at a given wavelength ($\lambda=1620$ nm). This was completed by varying the value of the effective index change in the range 0 to $\Delta N_{eff}=1.953e-5$, in nine steps. Simulation results for $s = \Delta I / \Delta N_{eff}$ are plotted in Fig. 3.9, showing a 7% change in output intensity as the effective index goes from zero to $1.953e-5$. The slope sign depends on the simulation wavelength, or the point of operation on the fringe pattern.

Figure 3.9: Simulated device sensitivity expressed with $s = \Delta I / \Delta N_{\text{eff}}$.

3.4 Summary

In this Chapter, modeling and simulation work has demonstrated the ability to predict and evaluate the interferometric sensing of optical devices with structured thin film deposited on sensing area. For a given set of geometrical parameters, the profile of a single mode Si_3N_4 waveguide was defined. This would be the building block for further modeling of optical devices such as segmented waveguides and Mach-Zehnder interferometer-based evanescent field sensors. The next step was the modeling and simulation of the dielectric structured thin film growth. Micro-structural growth orientation, film density, refractive index, film anisotropy, and the appropriate flux incidence angle were evaluated, providing valuable guidelines for the final device modeling as well as for the film fabrication process.

Finally, modeling and simulation of MZI with N_2 and CO_2 in the sensing environment was presented. Simulation results predict a 7% change in output

intensity as the gas content in the sensing environment changes from N_2 to CO_2 . In addition, higher sensitivity was predicted for devices with TiO_2 structured thin film deposited on the sensing layer.

Chapter 4

Fabrication

Processing and fabrication work presented in this Chapter concludes results obtained in Chapters 2 and 3 where guidelines for the waveguide technology, dielectric materials and sensing layer deposition technique, as well as the optical device design parameters were made available. Based on design parameters, fabrication involvement of this project aims to fabricate optical devices able to demonstrate the applicability of structured thin films for interferometric sensing.

In Section 4.1, a new technique for Si_3N_4 low temperature plasma etching [7] is established. Compatibility with integrated circuit process and the application of a carbon free etching based on a sulfur hexafluoride oxygen (SF_6/O_2) fluorine chemistry mixture in an electron cyclotron resonance (ECR) plasma source makes this method attractive to a wide range of applications such as integrated optics and MEMS devices. Concluding the purpose behind the development of this new etching technique, fabrication of optical devices based on Si_3N_4 waveguide is presented in Section 4.2. A partial etching of SiO_2 cladding layer established sensing areas into the wafer, thus preparing it for the final deposition of structured thin film. As shown in Section 4.3, dielectric thin films of different materials and orientations were deposited in stationary and rotating substrates. The film

deposition process was completed at Carleton University's fabrication facility and at the University of Alberta, an institution with a well known reputation and a broad range of expertise in the area.

4.1 Low temperature plasma etching of Si_3N_4

The etching process for the formation of Si_3N_4 channels in a waveguide should be anisotropic, to produce high aspect ratio features with vertical sidewalls, and highly selective to mask material, to minimize the erosion during the etching process. [86] As reported in Ref. [87], carbon-free etching of Si_3N_4 with $\text{SF}_6/\text{O}_2/\text{N}_2$ gas mixture in high-density ECR plasma source is possible. According to the authors, SiN_x etch rates of 20-30 nm/min have been achieved with high selectivity over both SiO_2 and Si. However, due to a poor selectivity of the process over the mask material, most of the experiments were limited to 2 min etching time, in order to avoid complete resist removal. Furthermore, no anisotropy was reported, making this process not ideal for Si_3N_4 waveguide fabrication.

In order to obtain anisotropic etching, the high chemical reactivity of atomic fluorine with silicon or nitride generally demands the use of a sidewall passivant or cryogenic temperatures. As demonstrated for polysilicon applications, SF_6/O_2 plasmas in which oxyfluorides provide sidewall passivation have achieved some success, [88] but are generally viewed to have insufficient anisotropy for waveguide applications. The use of fluorine chemistry at cryogenic substrate temperatures improves the process by further inhibiting lateral etching, [89] leading to high anisotropies with almost vertical sidewalls. A technique for successful etching of silicon with SF_6 plasma at cryogenic substrate temperatures was demonstrated, [90] and has since become widely used for SOI rib waveguide applications. [61] Several authors [91], [92] have studied the effect of different process

parameters such as sidewall angle, silicon etch rate, and selectivity to oxide and photoresist (PR) as a function of gas flow concentration as well as substrate temperature. In general, the results show that vertical-walled, anisotropic etching of silicon with good selectivity to oxide can be achieved.

As evident from applications to silicon, the aggressive cleaning action of sulfur hexafluoride with added oxygen makes this chemistry a good candidate for use in etching nitride waveguides. Nevertheless, there are problems that should be addressed: SF_6 is not only aggressive to nitride but also to PR. This low selectivity in the process limits etch time and etch depth of the nitride layer and also hinders formation of vertical sidewalls in the nitride ridge waveguide due to the erosion of mask material. The problem is worsened by the presence of oxygen in the plasma, as oxygen plasma etching can strip most solidified PR. Even a small percentage of fluorinated gas can greatly increase the removal rate for many resists. [93] Fluorine produces reactive sites on the polymer backbone, and also small amounts of fluorine increase the concentration of atomic oxygen in the plasma.

Based on these considerations, an integrated circuit compatible process to fabricate nitride optical waveguides is presented. Results shown in this work demonstrate that an SF_6/O_2 fluorine chemistry mixture in ECR plasma source and low substrate temperatures at about -30°C can successfully etch a $0.4\ \mu\text{m}$ deep nitride rib waveguide having width in the $2\ \mu\text{m}$ range. Furthermore, the combination of a hard 100 nm thick SiO_2 mask with a standard positive PR Shipley S1811 mask substantially improved etching parameters and increased allowable etching time. The process was optimized to obtain a good selectivity of nitride over the mask, with highly anisotropic etching, demonstrating nitride profiles with vertical sidewalls.

4.1.1 Experimental setup

Silicon starting substrates with $3.5\ \mu\text{m}$ thermal oxide were used. A low stress, low refractive index ($n=1.92$), PECVD Si_3N_4 film of $1\ \mu\text{m}$ thickness was deposited. The desired value of refractive index was achieved with the addition of hydrogen content on the deposition process. The thermal oxide layer serves as a buffer or underclad for the nitride waveguide core. Two different etch masks were considered: (a) a standard single layer positive PR S1811; and (b) a two layer mask with the first layer being a $100\ \text{nm}$ thick oxide, covered by a second layer of PR S1811. The only difference between these two mask configurations is the addition of the oxide hard mask, which should increase the process selectivity of Si_3N_4 over SiO_2 by acting as an etching barrier. [94]

Wafers with and without oxide-mask layer were processed. An additional lithography step was required for wafers having the $100\ \text{nm}$ SiO_2 mask layer. The pattern was transferred into the oxide layer by anisotropic dry etching, which was completed for 3 min in a reactive ion etcher (RIE) with trifluoromethane CHF_3/O_2 gas flows of 42.5/7.5 SCCM (SCCM denotes cubic centimeter per minute), respectively. Etched structures were investigated by optical microscopy, whereas the etch depth was measured with a Tenkor P1 profiler. The Si_3N_4 channels exposed for further plasma etching showed etch depth of $180\ \text{nm}$ ($100\ \text{nm}$ of SiO_2 and $80\ \text{nm}$ of Si_3N_4), leaving the rest of the wafer area covered by a two layer (oxide/PR) mask.

Plasma etching was completed using a PlasmaTherm 720 ECR etcher with a SF_6/O_2 chemistry and substrate cooling maintained by a temperature controller. Cooling was provided by a continuous flow of helium gas across the back side of the wafer. The helium was pre-cooled through a heat exchanger filled with liquid nitrogen. The wafer was clamped against an O-ring seal to prevent the escape of

the helium into the vacuum chamber.

The PlasmaTherm system was designed for use with 100 mm diameter wafers. However, fabrication experiments presented in this work were completed on 50.8 mm wafers or small pieces (typically 1x1 cm²). During the etching process in ECR, these small pieces were attached to a backing wafer using SPI Supplies double sided adhesive conductive carbon tape. The backing wafer and the attached sample were clamped to the cooled chuck, facilitating the cooling action. Gas flow rates into the chamber are mass-flow controlled. The flow ratio SF₆/O₂=3.65/2.21 SCCM was previously adjusted to maximize the etch rate and kept constant during the etching process. The operating pressure was 6 mTorr.

In addition to low temperature etching, several etching experiments were completed at room temperature. Samples were attached to the backing wafer with and without carbon tape, leading to two different etching temperatures. The purpose of this experiment was to investigate the effect of temperature on etch rate, anisotropy, and selectivity. After etching, the PR layer was ashed in oxygen plasma using a PlasmaPreen system, while the SiO₂ mask was left in place. Samples were examined in a JEOL JSM-6400 scanning electron microscope (SEM) operated at 20-25 kV.

4.1.2 Wafer and sample temperature measurements

The actual temperature of the sample is an important parameter, especially since low temperature plasma etching is involved in the experiments. In addition, the use of carbon tape to attach a sample on a cooled wafer introduces a temperature difference that might affect how the results can be applied to other situations. Therefore, an experiment to measure temperature on the surface of a dummy wafer and two samples (attached to the wafers with and without carbon tape) was set up.

Figure 4.1: Experimental setup for wafer and sample surface temperature measurement: (a) a thermocouple probe has been attached to the surface of each sample (upper and lower) and wafer (in the middle), with wires running to the six-pin port, and (b) a three-channel hand-held unit connected to the port making possible temperature readings from all three probes.

This would make possible a comparison between wafer surface temperature and the temperature of the sample piece with and without carbon tape. Photographs of the experimental setup are shown in Fig. 4.1, where Fig. 4.1(a) depicts the backing wafer and two samples attached to the wafer with and without carbon tape. The sample in the upper part of the picture has no carbon tape underneath, thus, polyimide tape (Kapton) was used to hold it in place. Three self adhesive type K thermocouple (TC) probes from OMEGA Corp. with temperature range -200 - 1250°C and standard limits of error 0.75% were attached to both samples and wafer surfaces as shown. A six-pin port on the ECR etcher [Fig. 4.1(b)] was dedicated for thermocouple wires to the hand-held unit (OMEGA HHM25-TC) connection, allowing temperature readings at all three channels.

The experiment was performed at chuck temperatures -31 and -81°C , with all other etching parameters as shown in Table 4.1. Plasma was turned "on" for 2 min; longer etching time was not advisable due to temperature limitations in TC

Parameter	Value	
Chuck temperature ($^{\circ}\text{C}$)	-30	21
Microwave power (W)	3	3
Microwave reflected power (W)	3	3
rf forward power (W)	10	10
Base pressure (Torr)	$1.5e - 6$	$1.5e - 6$
Operating pressure (mTorr)	6	6
SF ₆ /O ₂ gas flow (SCCM)	3.65/2.21	3.65/2.21

Table 4.1: Etch parameters for etching experiments at two different temperatures.

probe adhesives and connecting wires. Temperature reading was performed immediately after the etching plasma was turned off. Readings from chuck temperature controller, wafer surface, and both sample surfaces are summarized in Table 4.2.

As evident from the results, the temperature offset between these two attachment methods is large. Carbon tape adhesive with its high thermal conductivity provides an excellent path for heat dissipation toward the cooled wafer. There is no temperature difference between the carrier wafer and the sample attached with carbon tape, allowing the etching process to be at a truly low temperature. In contrast, there is a large offset between the temperature of the sample attached with Kapton tape and that of the carrier wafer. The thin evacuated layer between this sample and the carrier wafer acts as a thermal barrier, blocking heat flow. Also of interest is the temperature offset between the controller temperature readings and the wafer surface, which is due to the controller probe location being different from the location of the TC probe attached to the wafer surface. This offset is relatively small compared to the offset between the wafer surface and the sample without carbon tape and, as a result, it can be neglected. However, it becomes important and should be taken into account when samples attached

Measurement	Time	Temperature (°C)			
		Chuck	Wafer	Sample without C-tape	Sample with C-tape
1	Before etching	-31	-40.3	-30.4	-39.8
	After etching	-30	-38	+130	-37
2	Before etching	-81	-78	-69	-78
	After etching	-77	-80	+138	-77

Table 4.2: Temperature readings from chuck temperature controller, wafer surface, and sample surface for samples attached to the wafer with and without carbon tape.

with carbon are etched.

4.1.3 Low temperature plasma etching

Dry etching plasma process of Si_3N_4 rib waveguide dictates three main requirements: anisotropic etching with high aspect ratio to minimize the lateral under-etch, good selectivity to mask material, and high etch rate of nitride. In the following, all these requirements are investigated. The optimum parameters of the etching process were identified with preliminary experiments and are shown in Table 4.1; unless otherwise mentioned, these parameters were kept constant in all of the following experiments.

A sample with one layer of PR mask was patterned and attached to the backing wafer with carbon tape. The etching process was completed for 10 min at chuck temperature of -30°C . The addition of O_2 into the SF_6 gas flow makes anisotropic etching possible at wafer temperatures much higher than the -130°C reported in Ref. [90]. The aspect ratio obtained in the process is 5:1, resulting in

satisfactory anisotropy. A SEM image of a cleaved sample depicting a rib profile of Si_3N_4 waveguide with PR mask is shown in Fig. 4.2(a). For clarity, a schematic representation of the PR mask profile before and after etching as well as resist-nitride interface and undercut in the nitride rib is shown in Fig. 4.2(b). Of course, the SF_6/O_2 etching relies on sidewall passivation with oxide or oxyfluoride, forming a barrier in the sidewalls and limiting further etching. As a result, reasonably anisotropic etching with relatively vertical and smooth sidewalls was demonstrated. However, mask erosion and lateral undercutting around 80 nm are noticeable. This could be due to carbon compounds in the photoresist released in the process; these organics react to remove the sidewall passivation, giving lateral undercutting as shown in the rib profile.

Figure 4.2: A Si_3N_4 rib waveguide profile after 10 min etch in ECR with SF_6/O_2 gas chemistry and wafer temperature at $-30\text{ }^\circ\text{C}$; (a) SEM image of waveguide profile with 1 μm length scale, and (b) a schematic representation of the PR mask profile before and after etching.

Unfortunately, the addition of oxygen into the gas flow reduces the selectivity of nitride to resist, limiting etching time and potential depth of the trench. The mechanism is based on incorporation of atomic oxygen into polymer which weakens bonds between adjacent carbons. Oxygen also significantly increases the fluorine-free radical production in the plasma, hence increasing the resist etch rate as well as making the etch profile more isotropic. The remaining overall thickness and the highly slanted sidewalls of the PR layer of Fig. 4.2 demonstrate poor selectivity in the process. Measurement of the etch depth reveals 0.8:1 selectivity of Si_3N_4 over PR, with corresponding etch rates of 36 and 44 nm/min.

In order to improve selectivity in the process an oxide hard mask of 100 nm thick was deposited over the silicon nitride layer. Previous experimental work carried out on our ECR etcher with parameters consistent with this process has demonstrated selectivity of 4.6:1 of Si_3N_4 over SiO_2 . Therefore, a patterned oxide layer can serve as a hard mask for nitride etching. It should be noted here that at the end of the process, this oxide mask does not need to be removed; instead, it can serve as part of the oxide overcladding, a necessary step in further processing of buried waveguides. Etching of Si_3N_4 was performed for 8 min with plasma parameters shown in Table 4.1. After removing the resist, with the SiO_2 mask left in place, the overall etch depth was found to be 480 nm with etch uniformity $\pm 1.4\%$ measured across a 10 mm sample, achieving 300 nm Si_3N_4 etched in this process (recall that 180 nm was previously etched in RIE), with etch rate of 37.5 nm/min.

Figure 4.3 shows a SEM images of a cleaved waveguide. Verticality of the sidewall demonstrates highly anisotropic etching, with no signs of oxide-mask erosion or undercutting. Also, as expected, selectivity for Si_3N_4 over SiO_2 is very good, with the SiO_2 hard mask being very effective. However, due to our oxide

etching process not being very anisotropic, slanted edges at the SiO_2 cap of the rib profile can be seen. Fortunately, these slanted edges are limited to SiO_2 layer of thickness of 100 nm and do not affect the Si_3N_4 layer and the waveguide core itself.

Figure 4.3: SEM images of SiO_2 -masked waveguide (a) vertical sidewall demonstrating highly anisotropic etching, and (b) rib profile including 100 nm SiO_2 layer and 300 nm Si_3N_4 .

4.1.4 Temperature dependence of etching process

Further investigation of temperature dependent Si_3N_4 etching was completed through another etching experiment. Similar to the previous experiment, two patterned samples with oxide/PR mask were attached to the backing wafer: one with carbon tape adhesive (sample A) and one without any special adhesion or contact aid (sample B). While the chuck temperature controller was set at 21°C, the other parameters remain the same, as shown in Table 4.1. Under these conditions, etching was done for 8 min, with results shown in Fig. 4.4.

An image of a cleaved waveguide from sample A shows poor anisotropic etching

and 100 nm undercutting of Si_3N_4 , demonstrating a strong temperature sensitivity of the process. This predictable trend can be explained by desorption of the deposited inhibitor layers which increases with temperature, resulting in a negative dependence of inhibitor deposition with temperature. [95] The overall etch depth of 520 nm was measured, with Si_3N_4 etch rate of 42.5 nm/min. There is a 13.3% increase in the etch rate compared to the low temperature process.

Figure 4.4: SEM image of cleaved waveguide from sample A; rib profile shows around 100 nm undercutting.

Further, SEM images of waveguide ribs from sample B are shown in Fig. 4.5. A comparison between samples A and B reveals no qualitative difference in etching results. However, some quantitative differences can be noticed. Besides more undercutting, a profile measurement confirms greater etch depth (580 nm) and etch rate of Si_3N_4 (50 nm/min) for sample B. This can be explained by the higher sample temperature during etching. As previously demonstrated, the carbon tape adhesive applied on sample A achieves good thermal contact between sample and backing wafer, lowering sample temperatures. In contrast, sample B is characterized by a poor thermal contact, allowing elevated temperatures in the sample.

In general, these results once more demonstrate a strong temperature effect on

(a)

(b)

Figure 4.5: SEM images of a 2 μm wide waveguide rib from sample B: (a) waveguide butt and (b) cleaved edge.

the etching process. Low wafer temperature plays an important role on anisotropic etching. As a practical application, low temperature etching has demonstrated improved characteristics for Si_3N_4 waveguide etching.

4.2 Optical device fabrication

The procedure developed in the previous section was adopted to fabricate Si_3N_4 optical devices such as waveguides and Mach-Zehnder interferometers. Starting wafers with layer thickness and layer refractive indices are summarized in Table A.1 of Appendix A. Later in the process, sensing windows for structured thin film deposition were patterned. Two masks were involved in the process. The first mask, VOA1 already mentioned in Section 3, was used to pattern optical devices in a standard lithography process; it originated from McMaster University and was designed and fabricated as part of a joint research project between McMaster University, Optenia Corporation, and Carleton University. It contains straight waveguides, unbalanced, and balanced Mach-Zehnder interferometer structures with dimensions summarized in Table 4.3. The second mask (CU 27901A) was designed and fabricated at Carleton University fabrication facility using a Ledit mask editor and a mask generator. It contains openings $24\ \mu\text{m}$ wide and of three different lengths which align with straight waveguides, unbalanced, and balanced Mach-Zehnders. While openings on the Mach-Zehnder interferometer structures were intended as sensing windows, on the straight waveguides they would create sections where deposited nanostructured thin film and the mode propagating in the core would interact, allowing characterization of segmented waveguide optical properties.

In Table 4.4, fabrication processing steps are summarized, with more details given in Appendix B. While dry etching of SiO_2 hard mask and Si_3N_4 were

Parameter	Linear WG	Balanced MZI
Rib width (μm)	5	5
Y-splitting length (mm)	-	5.88
Linear section length (mm)	19.25	3.96
Active window length (mm)	3-5	3-5
Taper length (mm)	0.5	0.5
Distance between arms (μm)	-	200

Table 4.3: Dimensions of optical devices and sensing windows for future structured thin film deposition.

addressed in previous section, the additional steps required to complete the fabrication would be explained in the following.

The SiO_2 over-cladding layer was deposited at low temperature ($T=500^\circ\text{C}$) with atmospheric pressure chemical vapor deposition (APCVD) process in conventional furnace with SiH_4 gas and O_2 oxidant. After deposition, the oxide thickness of each wafer was measured with results shown in Table C.1 of Appendix C. There is a $\pm 10\%$ variation in oxide thickness from wafer to wafer depending on the wafer position in the quartz carrier, with deposition rate decreasing downstream the gas flow. However, by knowing the SiO_2 thickness on each wafer, etching time could be adjusted, thus addressing the issue of film non-uniformity. The SEM images of a cleaved waveguide with and without SiO_2 over-cladding are shown in Fig. 4.6.

Etching the SiO_2 over-cladding to create sensing wells was the next step in the process. Conventional buffered HF solutions used to etch a thermally grown SiO_2 were not recommended and should not be used for deposited SiO_2 films. Deposited SiO_2 films produced from the oxidation of silane etch much faster as compared to thermally grown SiO_2 . For this reason, wet etching with siloxide etchant was selected. It gives the necessary etch control for our deposited SiO_2 films.

Step	Process	Description
PS#1	Clean and dry wafer	Rinse (10 min) Bake (20 min) 200°C
PS#2	Photolithography	Mask R0109709 UV-expose (26 sec)
PS#3	Descum	O ₂ plasma etching (1 min)
PS#4	Etch 100 nm SiO ₂ mask	RIE (~130 sec)
PS#5	Etch 120 nm Si ₃ N ₄	ECR (~110 sec)
PS#6	Strip PR	O ₂ microwave oven (8 min)
PS#7	Pre-furnace cleaning	RCA (standard)
PS#8	Deposit 2 μ m SiO ₂	LoTOx quartz tube furnace
PS#1	Clean and dry wafer	Rinse (10 min) Bake (20 min) 200°C
PS#2	Photolithography	Mask CU 279 01A UV-expose (26 sec)
PS#3	Descum	O ₂ plasma etching (1 min)
PS#9	Wet etch SiO ₂	Siloxide Etchant (~13 min)
PS#6	Strip PR	O ₂ microwave oven (8 min)

Table 4.4: Fabrication steps for Si₃N₄ waveguide, including etching of SiO₂ cladding at sensing window.

(a)

(b)

Figure 4.6: SEM image of a cleaved waveguide (a) layer structure before etching SiO₂, and (b) after etching SiO₂, showing the Si₃N₄ rib.

Siloxide etchant is a modified HF buffered solution specific for deposited SiO_2 from silane processes. The etchant is formulated as a buffered system containing bifluoride where the HF activity is regulated by the equilibrium reaction between fluoride, bifluoride and HF buffer compounds. In addition, wet etching with siloxide etchant offers: (a) isotropic etching, avoiding vertical steps in the sensing wells, thus allowing a columnar film to be deposited, and (b) high etch selectivity of oxide over mask material and the underlying nitride. Experimental results for wet etching showed the etch rate of $1500 \text{ \AA}/\text{min}$, so the oxide layer of thickness $1.8\text{-}2.2 \text{ }\mu\text{m}$ required approximately 12-14 min of etching. As shown in Table C.1 of Appendix C, etching was completed for eight wafers with corresponding etching time and depth. A microscope image of an etched wafer depicting sensing wells with length 3.2, 4, and 4.8 mm is shown in Fig. 4.7.

Figure 4.7: A microscope image of a transparent over-cladding layer (with waveguides underneath) showing sensing windows where SiO_2 layer has been etched.

While a good care was taken to process all wafers under similar conditions, yet

results show some parameter variation such as the SiO_2 over-cladding thickness and the channel depth. These variations could be due to tolerance limitation in equipment used and also due to human inconsistency. However, simulation results have shown that the single mode condition on the fabricated waveguides should not be affected by these geometrical variations.

4.3 Deposition of structured thin films

Dielectric thin films were deposited with electron beam evaporation PVD technique. Three different oxides were considered and deposition was made on both stationary and rotating substrates. Deposition process was completed at Carleton University [6] and the University of Alberta fabrication facilities. Initially tilted Al_2O_3 films were deposited on glass substrates; preliminary data obtained from these films were beneficial for the following rounds of depositions.

4.3.1 Deposition on stationary substrates

A schematic representation of e-beam evaporation setup adopted for Al_2O_3 deposition is shown in Fig. 4.8. Stationary and obliquely inclined glass substrates with respect to the incoming vapor flux were positioned at a source to substrate distance (measured between centers) $d=33.8$ cm. Deposition was completed on two wafers at once, with angles between the wafer normal and the incident flux set at $\alpha=71^\circ$ and $\alpha=73^\circ$. The source diameter of 1.5 cm gives a small convergence angle of the incident flux at the substrate. The Al_2O_3 source material of 99.5% purity was provided by Cerac Inc. With a base pressure below 5×10^{-7} Torr maintained with a diffusion-pumped vacuum system, the material was pre-evaporated with a closed shutter for at least five minutes prior to deposition. As recommended,

Figure 4.8: Schematic representation of wafers obliquely positioned at flux incidence angles of 73° and 71°.

preconditioning consisting of slow sweeping of the electron beam to heat the top surfaces to white hot and fuse them was conducted. Evaporation parameters corresponding to Al_2O_3 deposition can be found at Cerac Inc. documentation and are not shown here. Deposition rate was maintained at 2-5 Å/sec with partial pressure of oxygen kept at 2×10^{-5} Torr.

After deposition, samples were prepared for SEM imaging. The image shown in Fig. 4.9 was taken with a field emission SEM ZEISS ULTRA 55. Samples underwent some preparation consisting on attaching another glass plate on the upper part of the Al_2O_3 film. Attachment was realized with an epoxy layer hardened on a hot plate for one minute. Furthermore, due to the dielectric nature of the sample, the phenomenon known as charging of the sample would be present. As the sample accumulates electrons from the high energy e-beam, it becomes a charged object, thus producing imaging difficulty. A well known solution to the charging problem is to simply coat the sample with a good electrical conductor. This was

done in a sputtering device where a thin layer of palladium-gold composition was deposited on the sample surface.

Figure 4.9: Image of Al_2O_3 film deposited at 71° . Layers from bottom to top are: glass substrate, Al_2O_3 film, epoxy and glass, with the last two being used for sample preparation only.

A magnified view of Al_2O_3 film reveals the image shown in Fig. 4.10, where film appears to have low to moderate porosity. This could be explained with low deposition angle, or the film could be more porous than it appears in the image, but the sputtered gold has covered voids in the sample. The actual film thickness as measured on the image is 887.0 nm.

The application of Eq. (3.3) to the above shown film demonstrates a reasonable agreement between predicted and measured values of angle β . As estimated from the SEM image, a flux incidence angle $\alpha=71^\circ$ has produced columns tilted at $\beta=48^\circ$ with substrate normal, whereas Eq. (3.3) predicts a $\beta=51.2^\circ$. Furthermore, similar results were obtained for other films deposited at $\alpha=73^\circ$ where the measured tilt angle $\beta=50^\circ$ is in reasonable agreement with the predicted one

Figure 4.10: SEM image of the Al_2O_3 porous film with glass substrate (bottom) and epoxy on top.

($\beta=52.3^\circ$).

On the next round, ZrO_2 was deposited on processed wafers. Wafer arrangement on the deposition chamber is shown in Fig. 4.11. This arrangement allows for the deposition on three wafers at once, with each wafer having columns tilted at $\beta=45^\circ$ with the wafer surface. However, the column orientation differs from wafer to wafer. Each wafer was offset differently, allowing columns to be at 0° , 45° , and 90° with the substrate flat or z -axes of light propagation direction. Because of this arrangement, we were unable to get a witness sample that accurately reflects the thickness of the actual films. Instead, witness samples (above and below the wafers) as shown in the figure were inserted. The actual thickness of the film on the wafers is most likely around the average between the thickness of the near and the far samples.

Deposition was completed onto unheated substrates with a source to substrate

distance $d=43$ cm and $85.0\pm0.1^\circ$ incidence angle. The evaporation source material (99.7% pure ZrO_2 from Cerac Inc.) was placed on a water-cooled copper crucible. Precondition, consisting on rapidly sweeping a low power electron beam to slowly and uniformly heat the surfaces of the material was completed before the evaporation process. During the evaporation, some amount of dissociation and oxygen loss was expected. The effect was reduced by providing sufficient oxygen backfill. The deposition pressure was maintained below 2×10^{-5} Torr by adding O_2 to the vacuum chamber.

Figure 4.11: Sample arrangement for deposition of slanted columns on three offset wafers (0° , 45° , and 90° with wafer's flat), including two samples above and below wafers for film thickness evaluation.

Images of ZrO_2 films with slanted columns shown in Fig. 4.12 were obtained using a Hitachi S-4800 field emission scanning electron microscope (FE-SEM). As pointed out, film thickness was measured on samples above and below the wafers, corresponding to far and near the source. Porosity of these films is evident with a true thickness of the films being about $1.42\ \mu\text{m}$.

(a)

(b)

Figure 4.12: Thickness of ZrO_2 films with slanted columns measured on witness sample located (a) above the wafers or far from source, and (b) below the wafer.

Titanium and oxygen form a number of stable phases, with the most practical being TiO , TiO_2 , Ti_2O_3 and Ti_3O_5 . All of these stoichiometries can be evaporated and subsequently oxidized to the final stable phase, TiO_2 , to yield a durable thin film coating. According to Ref. [96], there are advantages of using Ti_3O_5 as a source material as compared to other Ti-O combinations. By evaporating Ti_3O_5 as a starting composition, the only titanium species in the vapor is TiO , with the oxygen content remaining constant as the evaporation continues, leading to constant refractive index of the deposited film. On the contrary, evaporation of other phases may result in Ti-O combinations which change as the evaporation continues, thus resulting in films with non constant refractive index value. Based on this, Ti_3O_5 was used as a source material.

Deposition of 99.9% pure Ti_3O_5 (Cerac Inc.) was completed onto unheated substrates with a source to substrate distance of 43 cm and $85.0 \pm 0.1^\circ$ incidence angle. The evaporation source material was placed on a water-cooled copper crucible. When heated above their melting points, titanium oxides decompose into suboxides and liberate oxygen. To avoid outgassing and oxide spitting, a preconditioning process was run, consisting of the preparation of a homogeneous slug of material before beginning evaporation. Starting material was condensed into a homogeneous mass by melting at low power, using a swept beam. Oxygen evolved during this process, leaving a black, oxygen-deficient melt. After preconditioning, the melt was heated to evaporation temperature and the deposition process started. The deposition pressure was maintained around 3×10^{-6} Torr by adding O_2 to the vacuum chamber. Images of TiO_2 films taken with FE-SEM are shown in Fig. 4.13. Film thickness was measured on samples above and below the wafers revealing a true films thickness at about $1.38 \mu\text{m}$.

(a)

(b)

Figure 4.13: Thickness of a TiO_2 film with slanted columns measured on witness sample located (a) above the wafers or far from source, and (b) below the wafer.

4.3.2 Deposition on rotating substrate

Vertical posts can be considered as a special case of helical growth. Films of both ZrO_2 and TiO_2 were formed with substrate rotated slowly and continuously, relative to the deposition rate. For a deposition rate $0.25 \pm 0.05 \text{ nm/sec}$, rotation rate $\frac{dF}{dt} = 2 \text{ RPM}$ was maintained. With these parameters, the pitch of the helix is so small that it cannot be distinguished and thus the structure appears as a vertical column. The incidence angle of $85.0 \pm 0.1^\circ$ was set, providing required film porosity. No other change was made in the deposition system. Film images of both materials are shown in Fig. 4.14. As we can see, the structural details of these films depend on the source material being used. A comparison between these films reveals that TiO_2 , with a lower melting point, has higher surface diffusion and thus forms a better structure resembling vertical columns for the same substrate rotation speed.

Evaporated thin films of both materials (ZrO_2 and TiO_2), tend to be oxygen deficient. Previous studies [97] have found that annealing process at a low temperature for an extended period improves optical properties and crystalline structure. For this reason, films were oxidized in a furnace for 12 hours at 100°C .

4.4 Summary

An integrated circuit compatible process for Si_3N_4 low temperature plasma etching was established. Compatibility with integrated circuit process and the application of a carbon free etching based on SF_6/O_2 fluorine chemistry mixture in ECR plasma source makes this method attractive to a wide range of applications including MEMS devices. Results shown in this work demonstrate that an SF_6/O_2 fluorine chemistry mixture in ECR plasma source and low substrate temperatures

(a)

(b)

Figure 4.14: Films with vertical columns deposited on rotating substrates (a) ZrO_2 , and (b) TiO_2 .

at about -30°C can successfully etch a $0.4\text{ }\mu\text{m}$ deep nitride rib waveguide having width in the $2\text{ }\mu\text{m}$ range. This newly developed process was optimized to fabricate Si_3N_4 planar waveguides and optical devices in eight almost identical prototype wafers. A partial, wet etching of SiO_2 cladding patterned sensing areas for final deposition of structured thin film.

Dielectric structured thin film deposition was completed in two stages. Initially, the Al_2O_3 films were deposited on glass substrates, providing preliminary data and results valuable for the second round of deposition. In the second round of deposition, films of a specific material-nanostructure combination were deposited on eight wafers, resulting in different samples. Depending on the film material, samples can be divided in two groups, with TiO_2 and ZrO_2 . Samples of each group have films with vertical and slanted columns of different orientations. These samples will allow to investigate the effect of material/structure orientation on device sensing ability and on other optical properties.

Chapter 5

Optical Characterization of Thin Films

Thin film optical characterization is an important step in determining film properties and evaluating its potential applications. The work presented in this chapter approaches fabricated structured thin films of Al_2O_3 deposited on stationary, tilted glass substrates by means discussed in Section 4. Measurements on Al_2O_3 films were completed on early stages of this project, providing a set of preliminary data and directions which were essential in determining the final parameters of structured thin films.

Using ellipsometry, dielectric/optical properties of material, as well as porosity and its relation with the film refractive index and its dependence on deposition parameters, were explored. The refractive index determination of Al_2O_3 films in the visible range is demonstrated in Section 5.1. The optical model employed achieves a good matching with measurement data, providing accurate results on films of different porosities. In Section 5.2, optical properties of Al_2O_3 films such as birefringence and polarization are investigated. These optical properties play a major role in different applications such as optical sensing and optical component fabrication including polarizers, waveplates, and retarders. Results obtained

suggest that these films can be a very attractive alternative to materials that naturally exhibit birefringence. This low-cost process presents a great potential for fabrication of form-birefringent quarter-wave plates. In addition, by increasing the incidence angle during deposition process, almost reflection free coatings can be achieved.

5.1 Refractive index determination of Al_2O_3 structured thin film

In general, for any application of a material in optics it is essential to know its refractive index. A spectrophotometric method was implemented to determine the refractive index of Al_2O_3 structured thin films deposited on glass substrates. This method takes into account the layer thickness d , the refractive index n , and extinction coefficient k of the material. The relationship between the parameters that characterize the sample and the sample itself can be provided by spectrophotometric measurements, presenting a series of transmittances $T(\lambda)$ or reflectance $R(\lambda)$ of the sample in the spectral region of interests. The determination of layer parameters from $T(\lambda)$ cannot be achieved from the algebraic expression. An efficient method should be based on an iterative technique [98] with two key requirements: initial values should produce a solution somewhat close to the real one, and convergence to the solution should be provided.

A key element in satisfying these requirements is an optical model of a single (or multiple) layer film. Several models tested for the refractive index and extinction coefficient of Al_2O_3 tilted films revealed that the single layer model would better represent the measured data. The model includes a glass substrate and a dielectric film or parallel-sided slab of Al_2O_3 material. This model is often used for quick

calculations giving a good approximation for film optical properties. [98] In this way, n, k, d parameters can be defined from measured values of T , by completing the following steps: obtain experimental data for $T(\lambda)$, define the optical model of a single layer structure, apply Cauchy formula to obtain $n(\lambda)$ and $k(\lambda)$, calculate $T(\lambda)$ of the model from $n(\lambda)$, $k(\lambda)$, and d , and finally compare experimental versus model data and evaluate $n(\lambda)$, $k(\lambda)$, and d from $T(\lambda)$.

The optical parameters of the dielectric layer were modeled with the well known Cauchy approximation which can be expressed as:

$$n = a_0 + \frac{a_1}{\lambda^2} + \frac{a_2}{\lambda^4} \quad (5.1)$$

$$k = b_0 + \frac{b_1}{\lambda^2} + \frac{b_2}{\lambda^4} \quad (5.2)$$

$$N = n - jk \quad (5.3)$$

where a_0, a_1, a_2, b_0, b_1 and b_2 are the Cauchy parameters, N is the complex refractive index and λ is the wavelength.

However, the application of an optical model of a simple layer film is limited to isotropic and homogenous material, which is not the case with our highly porous columnar structured tilted films. To address this, the Bruggemann effective medium approximation shown in Eq. (3.4) was integrated into the procedure [99] and solved for the complex effective index N (instead of n_x). Using this equation, a porous Al_2O_3 film can be treated as one homogenous material with a complex effective refractive index N obtained after substituting the Cauchy approximation into N_d and N_p . Note that volume fractions for dense and pore materials (v_d and v_p) were simulated in Section 3.2.

With the assumption of a known substrate refractive index n_s and Eqs. (3.4), and (5.1-5.3), the optical transmittance of the model is obtained with Eq. (5.4).

A complete derivation for a single layer optical model can be found in Ref. [100].

$$T = \frac{2Y_0}{Y_0 m_{11} + Y_0 Y_s m_{12} + m_{21} + Y_s m_{22}} \quad (5.4)$$

where $\mathcal{M}$ is the characteristic matrix of the entire system that relates the fields at boundaries with coefficients:

$$\mathcal{M} = \begin{bmatrix} m_{11} & m_{12} \\ m_{21} & m_{22} \end{bmatrix},$$

n_0 and n_s are the refractive indices of air and glass substrate, and Y_0 , Y_s are optical admittances given with:

$$Y_0 = \sqrt{\frac{\epsilon_0}{\mu_0}} n_0 \cos \theta_{iI} \quad (5.5)$$

$$Y_s = \sqrt{\frac{\epsilon_0}{\mu_0}} n_s \cos \theta_{tII} \quad (5.6)$$

where ϵ_0 and μ_0 are permittivity and permeability of free space.

Transmission $T(\lambda)$ for different Al_2O_3 films deposited on glass substrates was measured with a Varian Cary 300 UV-Vis Spectrophotometer, with a wavelength range from 190 to 900 nm. The above mentioned procedure was applied to obtain the refractive index of a dense alumina film, deposited at incidence angle $\alpha=0^\circ$, as well as of two porous films ($\alpha=71^\circ$ and $\alpha=73^\circ$). A fitted model versus experimental data and the refractive index profile as function of wavelength for a dense alumina film of thickness $d=840$ nm are shown in Fig. 5.1. There is a good agreement between the refractive index obtained with our procedure and the values found in literature. [98], [101] However, small differences are due to deposition

conditions that vary broadly. The refractive index responds to high energy deposition techniques and to high substrate temperatures because both parameters decrease the void volume by increasing the packing density of the microstructure. Post-deposition baking in air can also raise the refractive index. Another factor affecting the refractive index is the amount of oxygen released in the chamber during evaporation process.

Figure 5.1: Refractive index of dense Al_2O_3 film of thickness $d=840 \text{ nm}$ deposited at incidence angle $\alpha=0^\circ$ (a) transmittance - experimental data vs. calculated, and (b) refractive index profile.

Experimental data and calculation results for a porous film $\alpha=73^\circ$ with porosity coefficient $f=0.42$ (58% of volume is bulk material) are shown in Fig. 5.2. Results demonstrate the effect of flux incidence angle on film porosity and refractive index. Similarly, another set of measurement data was obtained for a film

deposited at $\alpha=71^\circ$ with porosity coefficient $f=0.36$ (64% bulk material). To ease the comparison between three measured films, calculated refractive index profiles as function of wavelength are plotted on the same axes and shown in Fig. 5.3.

Figure 5.2: Refractive index of porous Al_2O_3 film of thickness $d=780$ nm deposited at incidence angle $\alpha=73^\circ$ (a) transmittance - experimental data vs. calculated, and (b) refractive index profile.

In general, higher deposition angle produces films with more voids and lower refractive index. This trend is demonstrated in our applications, where film deposited at incidence angle $\alpha=73^\circ$ has a lower refractive index. For deposited films, the refractive index appears to be in the range of 1.44-1.50; corresponding to deposition angle 71° - 73° . These values seem to be in complete agreement with simulated results shown in Table 3.1 where the refractive index $n=1.52$ was predicted for $\alpha=70^\circ$. The remarkable change in refractive index value $\Delta n=0.06$ that

Figure 5.3: Refractive index comparison for Al_2O_3 films of different porosities.

corresponds to a small change of deposition angle $\Delta\alpha=2^\circ$ can be attributed the critical angle of deposition, which appears to be around $\alpha=70^\circ$. [22] It is shown that film density decreases continuously with α , however with an increased rate near the critical angle, corresponding to a large change of Δn .

From the optical sensing standpoint, this fast decrease of film refractive index would be undesired; it increases the core-cladding refractive index difference, resulting in lower device sensitivities. By decreasing deposition angle, the refractive index issue could be solved, however, on the account of reducing film porosity and hence, the surface area. To conclude, such refractive index values obtained for Al_2O_3 structured thin films are not the desired ones, and for that reason were not qualified for the next round of deposition on Si_3N_4 waveguides. On the contrary, other dielectric materials considered such as ZrO_2 and TiO_2 with their higher refractive index, would be a better fit for our application.

5.2 Polarization properties of Al_2O_3 tilted films

Optical materials with anisotropic EM response are essential elements in polarizing optics applications such as linear polarizers, prisms, and waveplates also known as plate retarders. These components are typically constructed from precisely cut naturally occurring uniaxial and biaxial crystals to change the relative phase between two components of polarization. An inexpensive alternative to naturally occurring anisotropic materials which are generally incompatible with integrated photonic technologies are E-beam evaporated anisotropic films of materials such as Al_2O_3 , ZrO_2 and TiO_2 , [102] with TiO_2 offering a higher birefringence.

The anisotropic properties of Al_2O_3 films with highly orientated columnar structure result in optical birefringence. [103] When transmittance spectra were measured with two orthogonal directions of incident polarized light, in-plane birefringence, defined as the difference between the two in-plane refractive indices, produced refractive index differences Δn as large as 0.015. [103] The optical behavior of such anisotropic films was studied extensively [102]- [104], especially in applications similar to Al_2O_3 films shown in Section 4.3.1. These applications involve films that are optically biaxial with the birefringence available for light propagating normal to the substrate.

With these considerations, polarization properties of tilted Al_2O_3 thin films deposited on glass substrates are investigated. The SEM images on cleaved samples showed uniform column orientation with predicted tilt angles. Such columnar structure was expected to result in highly anisotropic and birefringent films, with optical axis oriented accordingly. Confirmation of polarization properties and optical anisotropy of these films is experimentally achieved with wavelength and orientation dependent optical power output. On overall, two porous films, namely DI-71 and DI-73 with corresponding thickness of 840 nm and 870 nm, and one

dense film, DI-0 of thickness 930 nm, were tested.

A common part of the setup for experiments described in this section is an optical system with two linear polarizers, a power source and a detector similar to Ref. [100]. The intensity of optical power would be proportional to the square of amplitude of electrical field and is given with

$$I = \epsilon c \langle E^2 \rangle \quad (5.7)$$

By introducing the second identical polarizer (analyzer) with optical transmission axis perpendicular to optical axis of the first polarizer, only the component $E_0 \cos \theta$ parallel to transmission axis of the analyzer will pass. As a result, the intensity of light reaching the detector will be a function of rotation angle between linear polarizers

$$I(\theta) = \frac{c\epsilon_0}{2} E_0^2 \cos^2 \theta \quad (5.8)$$

The maximum intensity, I_0 at $\theta=0$ would correspond to transmission axis of both polarizers being parallel, whereas the minimum value ($I_0=0$) would be at $\theta=90^\circ$. Thus the intensity can be expressed as

$$I(\theta) = I_0 \cos^2 \theta \quad (5.9)$$

The relations in Eqs. (5.7)-(5.9) suggest that by introducing a birefringent film (sample) in between two polarizers and rotating it in yz - and xy -planes, the state of polarization of the beam will change, allowing some light to go through the second polarizer. From the amount of light coming through we can determine the amount of rotation induced by the sample and relate that to film birefringence and thickness.

5.2.1 Wavelength dependent optical transmission spectra

A schematic representation of the experimental setup is shown in Fig. 5.4. A spectrophotometer was adapted to measure the transmittance of the samples. Light from an Oriel illuminator (model 7340) equipped with a 100W quartz tungsten halogen bulb (model 6333) was filtered using a motorized monochromator Oriel Cornerstone 130 (model 74000) controlled with TRACQ32 V3.1 control software. Monochromatic light was focused by a lens onto a cross-polarized set of linear polarizers. Another lens focused the light onto an Oriel silicon detector (model 70311) with transimpedance amplifier. The intensity of light delivered at the detector is measured by an Optical Power Meter (Oriel model 70310). The software makes possible a computer control output spectra of the monochromator and records the power as function of wavelength. A polycarbonate box painted black housed the focusing lenses, linear polarizers, and the sample holder placed in between polarizers, keeping the ambient light out of the detector during measurements.

Figure 5.4: Schematic representation of experiment setup for wavelength dependent optical power transmission.

Optical power transmission for polarizers in polarized state, crossed state, and crossed state with tilted film in between, versus wavelength in the interval 450-750 nm, is plotted in Fig. 5.5. Also, the effect of tilted film orientation on optical power transmission spectra can be observed from plots of Fig. 5.6. The reference transmission is given by the response measured when angle θ between the transmission axes of the analyzer and polarizer is zero and uncoated glass substrate is introduced. Next, the response was recorded when tilted film alumina coated substrate with optical transmission axes rotated by 45° in yz -plane was introduced. Finally, the third response was measured with the sample rotated at 45° in yz -plane and another 45° in xy -plane. The amount of optical power transmitted in both cases is expressed in percentage of the reference transmission, thus giving values relative to the direct signal measured in absence of the sample.

Figure 5.5: Optical power transmission for lenses in polarized state, crossed state, and crossed state with tilted film in between.

Figure 5.6: Ratio of optical power transmitted when tilted Al_2O_3 film coated substrate is introduced in between polarizer and analyzer lenses.

As can be seen, orientation of tilted film strongly affects the amount of optical power transmitted, demonstrating birefringence characteristics of the film. Measurements taken in oblique incident light were completed with extra care to avoid the influence of the anisotropy on optical behavior. As Fig. 5.6 shows, by introducing a properly aligned birefringent film, 6-14% of the reference output would reach the detector end. While the amount of light at the output depends on the wavelength, its dependence is not strong; demonstrating that the phase retardation action takes place even at wavelength $\lambda=700$ nm, which corresponds to the minimum in the curve. This demonstrates an advantage of these films; they can perform reasonably well for a wide range of wavelengths.

5.2.2 Orientation dependent optical power output

This experiment measures optical rotation angle ($\Delta\theta$) and azimuthal angle ($\Delta\Phi$) of deposited Al_2O_3 films. Source light is provided by a laser emitting $\mathcal{P}$ -state light at $\lambda=633$ nm. Initially, the system [104] was set up such that the polarizer and analyzer are in "crossed state"; no power at the detector. In this polarization state, detector calibration was performed and orientation angle of the analyzer was recorded. A tilted film coated glass substrate introduced in between polarizers with one of its principal axes aligned with one of the polarizers did not change the amount of light at the output. However, after rotating the sample by θ in zy -plane perpendicular to laser beam, some transmission occurs. Optical rotation angle was determined by rotating the analyzer by $\Delta\theta$, to remove any optical power at the detector. This compensates for the rotation caused by the optically active medium. The physical angle of rotation of the analyzer is then equal to the optical rotation $\Delta\theta$ caused by the medium.

Similarly, azimuthal rotation angle in xy -plane was found by rotating the film around z -axis by $\Delta\Phi$ until power at the detector reaches its maximum value. Measurement results for two samples DI-71 and DI-73 are summarized in Table 5.1. Light intensity at the detector as function of sample orientation angle θ is shown in Fig. 5.7, with measurements completed for clock wise and counter-clock wise rotation. Both sets of data show very similar behavior with optical power oscillating at $\approx 10 \mu\text{W}$. Also, optical power as function of azimuthal angle Φ was obtained and plotted in Fig. 5.8; optical power reaches its maximum at $\Phi=70^\circ$, demonstrating a strong relation. This plot provides an independent confirmation that columns are oriented around 70° from the plane of the sample.

The output power shown in Figs. 5.5-5.8 demonstrates a significant difference between the ordinary and projected extraordinary direction transmittances. The

Sample	Birefringence angle		Azimuthal angle		Structural angle	
	$\Delta\theta$	$\Delta P_{out} (\mu W)$	$\Delta\Phi$	$\Delta P_{out} (\mu W)$	α	β
DI-71	$4\pm0.2^\circ$	11	$50\pm0.2^\circ$	130	$71\pm1^\circ$	48°
DI-73	$4\pm0.2^\circ$	13	$67\pm0.2^\circ$	126	$73\pm1^\circ$	50°

Table 5.1: Experimental results for birefringence characterization of films with different flux incidence angle α .

Figure 5.7: Optical power output at the detector with variation of birefringence angle θ in zy -plane for azimuthal angle $\Delta\Phi=0^\circ$, where CCW and CW denote counter clock wise and clock wise rotation.

Figure 5.8: Optical power output at the detector with variation of azimuthal angle $\Delta\Phi$ in xy -plane for birefringence angle $\theta=48^\circ$. Note that measurement procedure was repeated three times with a corresponding data set shown.

film seems to be most transmissive along the columnar direction. By rotating the film in xy -plane perpendicular to the incoming polarized light, a $10\ \mu\text{W}$ variation of optical power at output can be observed. However, by rotating the film in xy -plane, or aligning the film columns to the incoming polarized light, the optical power at output was increased by an order of magnitude. Measurements were repeated three times, with results being consistent. They demonstrate that fabricated Al_2O_3 films behave like retarders, a well known optical component widely used to change the polarization of an incident wave. One of the two constituent $\mathcal{P}$ -states is somehow caused to lag in-phase behind the other by a predetermined amount. Upon emerging from the retarder, the relative phase of the two components is different than it was initially, and thus the polarization state is different as well.

With the direction of the optic axis parallel to the substrate surface, the $\mathbf{E}$ -field of the incident plane wave has components parallel and perpendicular to the optic axis, thus two separate plane waves propagate through the columns. [100] The crystalline orientation and columnar microstructure growth of our film makes them quite similar to other positive uniaxial crystals such as quartz. Therefore these separate plane waves travel at different speeds $v_{\perp} > v_{\parallel}$ experiencing refractive indices $n_e > n_o$ corresponding to extraordinary and ordinary axis. As a result, the o -wave moves across the film more rapidly than the e -wave. After traversing the film of thickness $d=840$ nm, the resultant electromagnetic wave is the superposition of the e - and o -waves, which now have a relative phase difference of $\Delta\varphi$. The relative optical path-length difference is given by

$$\Lambda = d(|n_o - n_e|) \quad (5.10)$$

and since $\Delta\varphi = k_0\Lambda$, the relative retardation would be

$$\Delta\varphi = \frac{2\pi}{\lambda_0} d(|n_o - n_e|) \quad (5.11)$$

As this equation suggests, the state of polarization of the emerging light depends on the amplitudes of the incoming orthogonal field components, whereas the relative retardation for a film with a given structure depends on the film thickness d . By changing the film thickness d , we can potentially make full-wave, half-wave, or quarter-wave plates in a very simple and inexpensive way.

Examining results of Table 5.1, the azimuthal angles of two measured films are different. This difference is due to the flux incidence angle α and column orientation β measured in Section 4.3.1, which for convenience are shown in Table 5.1. While β and $\Delta\Phi$ values are very close for DI-71 film, there is an almost

15° difference for DI-73. A nonlinear relationship β - $\Delta\Phi$ could be a possible explanation. While the alignment between the incoming polarized light and the column orientation is affecting the amount of elliptical polarized light off the film surface, it is not the main factor contributing to the film birefringence. The columnar structure orientation is only partially responsible for the birefringence, suggesting that the film density and more generally, the film packing condition is the other contributor. Beside the difference in $\Delta\Phi$, the other results do not consistently favor one or the other film. With equal rotation angles $\Delta\theta$ and inconsistent power measured at the detector, it is difficult to obtain an α - $\Delta\theta$ relation.

The optical rotation angle $\Delta\theta=4^\circ$ achieved in Al_2O_3 films was found to be quite respectable compared to values reported elsewhere, [41] for similar films. Given the film thickness of 840 nm, this angle represents an optical rotation of $4.76^\circ/\mu\text{m}$. In general, experimental results have shown that GLAD films can achieve an average rotary power ranging from $0.16^\circ/\mu\text{m}$ for MgF_2 to $1.05^\circ/\mu\text{m}$ for Al_2O_3 , [105] significantly less than values obtained by denser films which have demonstrated up to $5^\circ/\mu\text{m}$ for TiO_2 films. [41]

The enhanced rotation angle achieved in these Al_2O_3 films presents an attractive and quite inexpensive alternative technique for fabricating optical retardation plates of different dimensions. It is well known that crystal retarders require delicate cleavage process in their manufacture and become excessively costly for large-aperture configurations, while being fragile. Birefringent films of Al_2O_3 , as well as other dielectric materials, involve a much simpler fabrication process and can be easily deposited onto large areas. Tunable retarders, with different calibrations for different wavelengths, could be made by turning to advantage the gradual thickness variations of those films. Also, these birefringent films present an alternative to the conventional optical thin films that are based on the bulk

property of the film material. In contrast, Al_2O_3 films demonstrated here are based on the inside structure that can only be achieved in a thin film. The substantial function of the retardation is not caused by any inherent property of the bulk material but by the deposition process.

5.3 Summary

In this chapter, optical characterization of Al_2O_3 structured thin films deposited on stationary, tilted glass substrates, was presented. The experimental results and some theoretical interpretations addressed two main aspects of film optical properties: the refractive index and its relation with the flux incidence angle during deposition process, and film birefringence investigated through wavelength and orientation dependent transmitted light. Dielectric films analyzed induce significant asymmetry in the refractive index values that enable a retarder-like behavior. Quantitative analysis of the effect under normal incidence light at 632.8 nm was presented.

Using the ellipsometry and a single-layer optical model, the refractive index of films deposited at different flux incidence angles was determined. As results show, higher deposition angle produces films with more voids and lower refractive index. In agreement with predicted results, these measurements demonstrated a strong relation between flux incidence angle, film porosity and its refractive index. The refractive indices in the range of 1.44-1.50 were obtained, corresponding to deposition angles 73° - 71° . These values compare well with simulated results shown in Section 3.2 where $n=1.51$ was predicted for $\alpha=70^\circ$. Being in the range of the critical angle of deposition, a large $\Delta n=0.06$ was achieved for only 2° change in angle α . It is shown that film density decreases continuously with α , however with an increased rate near the critical angle.

A fast decrease of film refractive index with porosity would be undesired for our sensing application; it increases the core-cladding refractive index difference, resulting in lower device sensitivities. While a decrease in the deposition angle would have addressed the refractive index issue, it would have reduced the film porosity and the surface area, leading to reduced mode-measurand interaction. For this reason, Al_2O_3 structured thin films were not considered for further depositions on Si_3N_4 waveguide sensing devices.

Film birefringence investigation was completed by measuring the wavelength and orientation dependent transmittance of light propagating normal to the substrate plane. By introducing an Al_2O_3 coated glass substrate in between two crossed polarized lenses, the output power at the detector was increased by 14% ($\lambda=450$ nm) of the maximum power that corresponds to $\mathcal{P}$ -state optical signal measured in absence of coated substrate. The high birefringence of Al_2O_3 films is attributed to the columnar structure orientation and film density as well. This phenomena becomes more evident at depositions around critical angle $\alpha \approx 70^\circ$ with roughly 65% packing density. Results suggested that the birefringence cannot be essentially related to the inclination of the columnar structure. However, it can be attributed to the form birefringence caused by the packing condition of the columns in an obliquely deposited thin film.

Experimental results obtained for orientation dependence transmittance allowed the polarization rotation angle to be evaluated, which in turn permitted birefringence estimation. As demonstrated, a film of thickness 840 nm achieved a 4° polarization rotation angle, or $4.76^\circ/\mu\text{m}$. Arguably, such optical rotation can be considered amongst the highest values reported in the literature. By rotating the substrate in a plane vertical to the propagating beam, the optical power at the output oscillated from 0 to 11 μW . Further, by aligning the columns with the

incident beam ($\Phi=68^\circ$), the output power was increased to a maximum 130 μW .

The enhanced rotation angle achieved in these Al_2O_3 films presents an attractive and quite inexpensive technique for fabricating optical retardation plates of different dimensions. Tunable retarders, with different calibrations for different wavelengths, could be made by turning to advantage the gradual thickness variations of those films. The columnar structure of obliquely deposited films suggests that similar behavior should be observed in films of virtually any material, where the substantial function of the retardation is not caused by any inherent property of the bulk material but by the deposition process.

Chapter 6

Component Characterization and Application to Optical Sensing

The optical measurements presented in this chapter aim to validate the newly proposed platform for interferometric-based sensing applications. Results obtained here will conclude the overall simulation and fabrication work demonstrated so far in this project. In Section 6.1, the experimental setup for optical device characterization is outlined. Optical devices measured and characterized include linear and segmented waveguides as well as MZI for sensing applications, with measurements completed on both transmission spectra as well as time response of MZI output. In Section 6.2 the optical loss analysis of fabricated waveguides is presented; it starts with simple waveguides with SiO₂ over-cladding layer of thickness 1.8 μm and continues with segmented waveguides where SiO₂ over-cladding was removed in an area 24x3100 μm^2 above the ridge and replaced by ZrO₂ or TiO₂ structured thin film of different configurations. An anomalous wavelength response observed in segmented waveguides is analyzed in Section 6.3. Modal analysis completed on a slab waveguide revealed that deposited TiO₂ films have high birefringence properties with a $\Delta n=0.044$ between TE and TM modes.

In Section 6.4, optical characterization of fabricated MZI is presented. The

sensing ability of devices with ZrO_2 and TiO_2 films in the sensing area is demonstrated in Section 6.5, where the refractive index of CO_2 is measured and the sensitivity of devices with different film configuration is evaluated.

6.1 Experimental setup

Optical device testing was performed at the National Research Council (NRC) optical testing laboratory. A schematic of the measurement setup is shown in Fig. 6.1. An Agilent Lightwave Measurement System 8164B with tunable laser source module 81600B 200 and wavelength range 1440-1640 nm was used as the light source. The signal from the laser source is transmitted through a single mode polarization-maintaining (PM) fiber into a polarization rotator controller which manipulates and controls the state of polarization of the input beam of light and couples the adjusted light into an output fiber. This polarization controller consisted on three components, a polarizing glass filter for TE or TM polarized light output, a half-wave plate, and an output coupler with fiber pigtail. The half-wave plate position is controlled by a universal motor controller/driver (ESP 300 by Newport) via a stepper motor, to allow precise and repeatable polarization rotation for TE or TM selection. It also allows the alignment of light as closely to the proper PM fiber axis as possible, thus maintaining the state of linear polarization and orientation of light along one of principal transmission axes within the output fiber.

A single mode PM tapered fiber was used to transmit the linearly polarized optical signal from polarization controller output into the device under test (DUT). Butt-coupling of fiber end to the waveguide facet provides good efficiency and mode matching ability, ensuing minimum coupling losses. The output light from

Figure 6.1: Optical measurement setup at NRC optical testing laboratory.

the sample is collected using a 20x objective lens and passed through an analyzer consisting on a broad-band polarizer and a polarizing cube beam-splitter which splits the beam into two orthogonal, linearly polarized components. The output power is directed and measured with an Agilent 81624B InGaAs detector, or reflected (through a mirror) to an infra-red camera (Find-R-Scope 1800 nm) for visual inspection and alignment facilitation. The position of the input tapered fiber and the output lens can be controlled with individual stages (MG 17MAX303), whereas the sample can be securely placed in a vacuum controlled stage (17ANF005), providing steadiness during the measurement process. Wavelength and/or time response measurements were run by LabView software.

To ensure reliable performance of a system it is important that overall system losses without the DUT are measured. The ratio of the input power level and the power transmitted and detected at the output end gives an overall system loss, which later can be used as a reference in quantifying waveguide propagation losses. With the input power $P_{in}=1$ mW injected into the fiber from the optical source, the optical output power P_{out} received at the detector was measured and calculated $\text{dBm}=10\log_{10} P_{out}$ for linear polarized light at input-output with combinations:

TE-TE, TM-TM, TE-TM, and TM-TE. Results of Fig. 6.2 demonstrate that both TE-TE and TM-TM are transmitted with low losses (-3.2 dBm) and more than -22 dBm cross polarization.

Figure 6.2: Reference losses for the setup of Fig. 6.1.

6.2 Characterization of Si_3N_4 rib waveguides

Transmittance measurement and loss analysis of a waveguide are important for optical characterization. Losses due to light coupling in/out the waveguide and also scattering and absorption in the waveguide itself do exist. In the following, some of these losses are measured and analyzed. The analysis starts with a simple waveguide with SiO_2 over-cladding of thickness $1.8 \mu\text{m}$ and continues with segmented waveguides, where SiO_2 over-cladding was removed in an area $24 \times 3100 \mu\text{m}^2$ above the ridge (Fig. 4.7), and replaced by ZrO_2 or TiO_2 structured thin film of different configurations as described in Section 4.

6.2.1 Waveguides with SiO₂ over-cladding

With the setup described in Fig. 6.1, waveguide transmission spectra for TE-TM and TM-TM polarization were measured. After subtracting reference system losses, a typical wavelength response for each polarization is plotted in Fig. 6.3. As we can see, while the attenuation is low for wavelengths $\lambda > 1580$ nm, there is a large dip on the transmission spectrum covering a broad range of wavelengths (1470-1580 nm) with a minimum value of -25 dBm at 1520 nm. In order to explain this behavior a detailed loss analysis would be required, aiming to evaluate the contribution of different loss components present on the waveguide material and structure.

Figure 6.3: Signal attenuation on WG00 for TE and TM polarization.

Coupling losses would be at fiber-to-waveguide connection. Those are mainly extrinsic coupling loss, increased by sources such as: (a) reflection losses at the facet (Fresnel loss), (b) lateral/angular misalignment, (c) fiber and waveguide core

dimension mismatch, (d) numerical aperture mismatch, (e) refractive index profile difference, and (f) optical scattering at waveguide facet. These types of losses can be substantially reduced by following proper alignment procedures.

Fresnel reflection loss is one of the most basic forms of loss. It occurs as a result of the reflection in air-semiconductor interface and is associated with the discontinuity or impedance mismatch at interfaces between two media where there is a step change in the refractive index. Quantitatively, it is the ratio of incident over reflected power at the facet expressed in dB. Fresnel loss (FL) at the interface can be obtained with:

$$FL = -10 \log(1 - r) = -10 \log\left[1 - \frac{(n_a - n_b)^2}{(n_a + n_b)^2}\right] \quad (6.1)$$

where r is the reflected power, n_a and n_b are the refractive indices of air and waveguide core, respectively 1 and 1.92. Calculation of Fresnel loss gives $FL=0.46$ dB/facet, totaling 0.92 dB.

While it is easy to calculate the Fresnel loss, the situation is different for the others (b-f); they strongly depend on skills, sample preparation techniques, and measurement setup. Therefore, a qualitative analysis would better serve our purpose. Mode mismatch between the input fiber and the waveguide core is caused by the mode size difference. This mode mismatch reduces coupling efficiency. Also, the quality of the waveguide facet depends on cleaving. Since no facet improvement technique was employed, it is expected that rough waveguide facets will introduce some optical scattering. However, with the proper care taken, coupling losses were maintained at a low level. Their contribution was estimated at no more than 3.5 dB in total.

However, the system and coupling losses as described cannot be blamed for the

large dip in the transmittance; their dependence on wavelength is weak. What remains is the waveguide itself, with material properties and structure both strongly affecting light scattering and/or absorption in the waveguide. Basically, scattering losses are caused by the interaction of light with density fluctuations within a waveguide. An IR camera image (Fig. 6.4) of the waveguide, as light at $\lambda=1555$ nm propagates through, shows no signs of significant scattering, leaving the absorption as the main cause for such high losses.

Figure 6.4: IR camera image (from above) of a 19 mm long waveguide as light at wavelength $\lambda=1555$ nm propagates in it.

Absorption in a waveguide material could be due to imperfections in the atomic structure, and/or the intrinsic/extrinsic properties of the material. As demonstrated, [106], [107] the intrinsic properties of Si_3N_4 are the main reason behind this large absorption in the NIR region. The main cause of this intrinsic absorption is the characteristic vibration frequency of atomic bonds of silicon-hydrogen (Si-H) and nitrogen-hydrogen (N-H). The interaction between the vibrating bonds and the electromagnetic field of the optical signal causes intrinsic absorption. Light energy is transferred from the electromagnetic field to the bond.

Attenuation coefficient α (dB/cm) of a typical waveguide was obtained with the application of the "cutback" technique, [108] with measurements completed at the Carleton University's optics laboratory. Transmittance spectrum of a waveguide at two different lengths (16 mm and 13 mm) was measured with results summarized in Fig. 6.5. From several sets of data available, the average values at each wavelength point was calculated, producing two averaged transmittances (one per each length).

Figure 6.5: Transmission spectra (as measured) for four different waveguides of lengths of 16 and 13 mm.

Transmittance at $\lambda=1.55 \mu\text{m}$ was extracted for both waveguide lengths and plotted on Fig. 6.6. The slope of the graph yields the waveguide attenuation coefficient $\alpha=7.1 \text{ dB/cm}$. Such value is in complete agreement with other published works [109] where the typical attenuation of a PECVD amorphous silicon nitride thin film is high at $1.4\text{-}1.6 \mu\text{m}$ wavelength range with a peak value of 7.5 dB/cm at $1.52 \mu\text{m}$. However, the attenuation becomes much lower in the $0.63\text{-}1.35 \mu\text{m}$ or

in the NIR region.

Figure 6.6: Transmittance measurement as a function of sample length using "cutback" technique; slope of the graph gives attenuation coefficient $\alpha=7.1$ dB/cm at $\lambda=1.55 \mu\text{m}$.

Having identified the attenuation coefficient, coupling and system losses for this setup were obtained. Another transmittance measurement for a wider wavelength range was carried out on the 13 mm long waveguide. After subtracting coupling and system losses for this setup from the transmittance measured, the plot of Fig. 6.7 was obtained. For comparison, transmittance measured from NRC setup is also plotted. While there is a very good agreement in the wavelength range of 1530-1555 nm, some discrepancies are noticeable outside this range. The reason is that the assumption of a constant attenuation coefficient as calculated at $\lambda=1555$ nm does not hold for the entire range, but is a function of the wavelength and gives a correct representation at the wavelength where it was calculated. Using this value outside the range would of course introduce some inaccuracies.

According to literature recommendations, Si_3N_4 waveguide attenuation and

Figure 6.7: Attenuation for different waveguides measured at NRC and Carleton University.

its refractive index largely depend on the amount of hydrogen included in the film during the deposition process. As is the case for our waveguides, the core material was fabricated with PECVD technique where some amount of hydrogen was incorporated to obtain a low refractive index film. Several works [59], [106] were found to support the idea that absorption at telecom wavelength can be reduced by annealing samples at 1150°C for one hour. Following this, annealing treatment was completed on test wafers in a conventional furnace with N₂ gas environment. Several runs at different temperatures were made to find the appropriate settings, with results summarized in Table 6.1. As we can see, for temperatures 800°C and higher, visible cracks on the film and substrate warping was observed. However, annealing at lower temperatures for a longer period showed no structural damages on the film.

Run	Temp. (°C)	Time (min)	Results & comments
1	1150	60	Substrate warping and cracks
2	800	90	Visible cracks, no warping
3	600	120	Neither warping nor cracks

Table 6.1: Annealing temperatures and results for wafers with Si_3N_4 film of thickness $1\ \mu\text{m}$.

With $600^\circ\text{C}/120\ \text{min}$ temperture/time settings, annealing process was completed on several waveguides. Transmittance spectrum was measured for the samples which underwent a non-destructive annealing treatment. Unfortunately, there was no improvement on the waveguide attenuation, demonstrating that annealing process at 600°C was not effective. The hydrogen still remains trapped in the waveguide structure. This deduction was found in agreement with results obtained for silicon oxy nitride. [106]

While the high waveguide attenuation at $\lambda=1.48\text{-}1.56\ \mu\text{m}$ was due to intrinsic material absorption, waveguides perform much better outside this range. To demonstrate, insertion loss (IL) of a typical waveguide at $\lambda=1.62\ \mu\text{m}$ were found to be 8.3 dBm; after subtracting $FL=0.92\ \text{dBm}$ obtained with Eq.(6.1), and system losses of 3.2 dBm from Fig. 6.2, the remaining value of 4.28 dBm would present losses due to mode mismatch and waveguide material absorption, inhomogeneity and surface roughness.

A comparison with simulation results reveals a small difference of 0.62 dB between measured and simulated losses. The simulated power at the output calculated with the power overlap integral with the fundamental mode estimated 3.56 dBm losses due to the mode mismatch only, without taking into account any absorption or scattering effect. As a result, the difference between measured

and simulated losses would present losses due to absorption or scattering in the fabricated waveguide. Of course, such low losses obtained at this wavelength demonstrate high quality of fabricated waveguides, with low intrinsic absorption and scattering due to inhomogeneity and surface roughness. The fabrication process employed on the samples achieved smooth core-cladding interface. Absorption losses due to high hydrogen content on the Si_3N_4 layer can be overcome by operating the device at wavelength $\lambda=1.62\ \mu\text{m}$.

6.2.2 Optical characterization of segmented waveguide

Segmented waveguide characterization is an essential task in the development of photonic integrated circuits for a variety of applications, including sensors [2] and next generation optical interconnects. Transmission spectra for segmented waveguides similar to one shown in Fig. 6.8, with a 2 or 3 mm long segment of ZrO_2 or TiO_2 structured thin film were obtained. The refractive properties of a biaxial film determined by three principal refractive indices, n_1, n_2, n_3 , and the location of the principal axes to which these indices are referenced, [23] are expected to affect the mode behavior in general and the optical power output in particular. Result analysis and a comparison between film materials and structures, as well as the effect of nanostructured cladding on waveguide evanescent field and polarization properties is presented. Light intensity at waveguide output can be correlated with features in the evanescent fields that may prove useful for waveguide sensors.

The analysis presented here is based on assumptions that: tolerances in the waveguide length and fabrication are small; the input/output facets are in similar conditions; and the absorption loss in nitride core is similar in all waveguides. With these assumptions, scattering and absorption losses and evanescent field coupling into the structured thin film segment are expected to play the main role

Figure 6.8: Cross section along propagation direction of a segmented waveguide with anisotropic film deposited on the sensing window, showing location of principal axes in a biaxial thin film model.

in differentiating between waveguides.

Measurements were completed on eight samples, divided into two groups named WG00 and WG01. Each group has four waveguides with four different dielectric film orientations in the segment. The notation exercised to identify each waveguide is as follows: the first letter (Z or T) stands for ZrO_2 or TiO_2 , the following characters stand for column orientation with the beam propagation direction, where 0, 45, and 90 are the offset angles of 45° slanted columns, and V is for vertical columns. Measurements were completed for input-output linearly polarized light in both TE-TE and TM-TM configurations. The wavelength range 1580-1640 nm was considered.

Measurement results for waveguides with ZrO_2 cladding in the segment are shown in Figs. 6.9 and 6.10. A comparison between TE and TM polarized light transmitted shows that TE orientation is propagated with lower losses. This is confirmed in both waveguide groups WG00 and WG01. Explanation for this behavior is based on the field orientation and its interaction with the structured thin film deposited in the segment. As it propagates, the TM orientation goes deeper into the cladding layer. Of course, this is the reason why TM orientation

is more sensitive to environment changes in the cladding layer, and as a result is commonly used for sensing applications. However, with the specifics of our film, this higher interaction results in higher scattering and absorption of light into the film. Also, linearly polarized light with TM orientation has better coupling into the film columns with more light propagated along the columns, thus, being another contributing factor that increases losses.

Furthermore, for a given linearly polarized light, either TE or TM, the film structure and column orientation affects transmission spectra. This becomes apparent when comparing graphs on the same plot, where films with vertical columns (ZV) demonstrate higher losses as compared to films with 45° slanted columns (of all three orientations). These results are consistent on both waveguide groups. This could be related to a higher scattering loss due to the 90° angle between propagation direction and vertical columns.

A comparison of samples with slanted columns reveals no consistent results, with all three averaging similar losses. This makes it difficult to distinguish and speculate on which structure would be the favored one for future consideration, and for this reason all three would be treated equally.

Measurement results for waveguides with TiO_2 structured thin film deposited on the segment are summarized in Figs. 6.11 and 6.12. As confirmed in both waveguide groups (WG00 and WG01), the TE linearly polarized light is transmitted with much lower losses than TM polarization, with a difference being in excess of -12 dBm. Explanation for this behavior is based on the field orientation and its interaction with the deposited structured thin film layer. As it propagates, the TM polarized light goes deeper into the TiO_2 layer; evanescent part of the field is larger, resulting in more interaction with the film. However, with the specifics of our film, this higher interaction results in higher scattering and absorption of

Figure 6.9: Transmission spectra for waveguides WG00 with ZrO_2 cladding (a) TE-TE, and (b) TM-TM polarization.

Figure 6.10: Transmission spectra for waveguides WG01 with ZrO_2 cladding (a) TE-TE, and (b) TM-TM polarization.

light into the medium.

For a given linearly polarized light, either TE or TM, the film structure and column orientation affects transmission spectrum. This becomes apparent when comparing graphs on the same plot and also in between plots. For TE polarized light, both waveguides demonstrate that devices with columns at 90° offset (T90) have lower losses, whereas devices with columns at 0° offset (T0) have the higher loss of all. However, this standing is not true for TM polarized light [Figs. 6.11(b) and 6.12(b)], where T90 devices have by far the highest loss of all, and T0 devices perform reasonably well. This behavior could be in part due to the polycrystalline nature of rutile TiO₂ film and its columnar orientation during the deposition process. However, a more detailed analysis of this factor and its effect on the wavelength dependent transmission spectra observed for TE and TM polarizations on specific devices would be presented in the next section.

Based on this analysis, some deductions can be drawn. Starting with polarization of transmitted light, results show that TE light propagates with lower loss than TM, with an average difference of approximately $\Delta P_{out} = -4$ dBm for devices with ZrO₂ film and $\Delta P_{out} = -12$ dBm for devices with TiO₂ films. While for TE, losses are comparable for both materials, a higher TM loss for TiO₂ might relate to (a) high anisotropy of the film caused by material polycrystalline orientation, and (b) the fact that film refractive index is closer to the core refractive index value. These factors contribute to changing the modal profile by pulling the evanescent field deeper into the cladding layer, thus increasing both the interaction and the amount of scattering loss into the layer.

Waveguides WG00 and WG01 demonstrate similar losses even though the segment length is different, proving that section length does not strongly affect waveguide loss. However, it should be noted that more data (lengths) would

(a)

(b)

Figure 6.11: Transmission spectra for waveguides WG00 with TiO_2 cladding (a) TE-TE, and (b) TM-TM polarization.

(a)

(b)

Figure 6.12: Transmission spectra for waveguides WG01 with TiO_2 cladding (a) TE-TE, and (b) TM-TM polarization.

be required to explicitly obtain a section length-loss relation. A comparison in between film orientations reveals: (a) for devices with ZrO_2 film, all three orientations with 45° slanted columns perform reasonably well, however, devices with vertical columns demonstrate the highest loss of all cases and (b) for devices with TiO_2 film, T90 orientation performs better for TE polarized light, whereas T45 performs better for TM polarized light.

To conclude, mainly TE polarized light would be considered for the future measurements. In addition, some measurements with TM polarization would be considered when sensing ability is to be evaluated. Because the TM is expected to be more sensitive to environment changes on the sensing region, strength of the output signal might be of lesser importance, thus permitting application on sensing devices.

6.3 Optical constant determination of TiO_2 films via modal analysis

As pointed out in the previous section, segmented waveguides with TiO_2 film, and in particular those with columns orientated at 0° and 90° offset exhibit quite strange behavior. As shown in Fig. 6.13, light at the output of waveguide T0 for TE polarization oscillates with almost 5 dBm peak-to-peak amplitude, whereas the TM polarization shows no such oscillations. On the contrary, for T90, the output for TE shows no oscillation, whereas the TM is almost cut-off.

The highly birefringence properties of polycrystalline TiO_2 films deposited by GLAD technology could be the reason behind this behavior. The difference in refractive indices for TE and TM polarized light propagating in the segmented

(a)

(b)

Figure 6.13: Transmission spectra for waveguide WG00 with TiO₂ film with slanted columns at (a) 0° offset, and (b) 90° offset.

waveguide may affect the number of modes in this highly asymmetric slab waveguide. While the slab waveguide was designed to be a single mode, it may support more than just the fundamental mode for TE polarization, but still be single mode for TM polarized light. However, a more detailed analysis focused on guided modes in a basic slab waveguide with a dielectric film in the cladding layer would be required in order to support this speculation.

A side view of the slab waveguide has been sketched in Fig.6.14. The thin film structure consists of three different materials, one for the substrate, one for the film, and the third for the cover. Characteristic parameters considered are the refractive indices n_s, n_f and n_c of the substrate, the film, and the cover, the film thickness h , and the free-space wavelength λ or the propagation constant $k=2\pi/\lambda=\omega/c$ of the light. Let us assume that light in the waveguide propagates in the z -direction, that confinement occurs transversely in the x -direction, and that both the structure and the light are uniform in the y -direction perpendicular to xz . For a guided mode of the slab waveguide, the self-consistency condition (also known as "the transverse resonance condition") [110] can be expressed as:

Figure 6.14: Side-view of a slab waveguide showing propagation corresponding to a guided mode.

$$2kn_fh \cos \theta - 2\phi_s - 2\phi_c = 2\nu\pi \quad (6.2)$$

where ν is the integer (0,1,2,...) which identifies the number of modes, and the phase shifts ϕ_s and ϕ_c are a function of the angle θ .

Following the procedure in Ref. [110], a numerical evaluation of this equation gives the ω - β diagram for the asymmetric slab guide. First, a normalized frequency and film thickness V are defined by

$$V = kh\sqrt{n_f^2 - n_s^2}, \quad (6.3)$$

and then a normalized guide index b with:

$$b = \frac{N^2 - n_s^2}{n_f^2 - n_s^2} \quad (6.4)$$

where N is the effective guide index given with $N=\beta/k$ and β is the propagation constant. The index b is zero at cut-off and approaches the unity far away from it. Finally, a measure of the asymmetry of the waveguide structure is introduced

$$a = \frac{n_s^2 - n_c^2}{n_f^2 - n_s^2} \quad (6.5)$$

This measure applies for TE-modes and is close to infinity for strong asymmetry ($n_s \neq n_c$) such as our case.

For TE modes, using the normalization equations (6.4)-(6.5), the dispersion relation of Eq. (6.2) can be written:

$$V\sqrt{1-b} = \nu\pi + \arctan\sqrt{\frac{b}{1-b}} + \arctan\sqrt{\frac{b+a}{1-b}} \quad (6.6)$$

The situation for the TM modes is analogous, but not quite as simple as for

the TE modes [110], with the asymmetry measure defined

$$a = \frac{n_f^4 n_s^2 - n_c^2}{n_c^4 n_f^2 - n_s^2} \quad (6.7)$$

$$d = (1 - \frac{n_s^2}{n_f^2})(1 - \frac{n_c^2}{n_f^2}) \quad (6.8)$$

and a reduction factor q_s given by

$$q_s = \frac{n_s^2/n_f^2}{(1 - b) + bn_s^4/n_f^4} \quad (6.9)$$

As we can see, there are three parameters needed to represent a general waveguide structure. However, each one of these parameters a , d , and q depends only on the index ratios n_s/n_f and n_c/n_f . As shown in Ref. [110], calculation of the $b-V$ curve for TM modes can be simplified by considering only a and n_s/n_f and neglecting the term bd by assuming $b=0$. The error in calculation will be less than 1%. The normalized form of the dispersion relation for the TM modes becomes

$$V(\sqrt{q_s} \frac{n_f}{n_s})\sqrt{1-b} = \nu\pi + \arctan \sqrt{\frac{b}{1-b}} + \arctan \sqrt{\frac{b+a(1-bd)}{1-b}} \quad (6.10)$$

A numerical evaluation of Eqs. (6.6) and (6.10) yields the normalized ω - β -diagrams shown in Fig. 6.15 where the guide index b is plotted as a function of the normalized frequency V for four different values of the asymmetry measure $(0,1,10,\infty)$ and for the mode orders $\nu=0,1$ and 2 for TE mode and, the asymmetry measure $a=1$, $n_s/n_f=0.7, 0.76, 0.8$, $d=0$, and $\nu=0,1$ and 2 for TM modes.

With the normalized frequency $V=7.83$ corresponding to our waveguide, the normalized guide index b was evaluated from plots of Fig. 6.15. The asymmetry measure that applies to our waveguide was calculated with Eqs. (6.6) and

Figure 6.15: Guide index b as a function of normalized frequency V for the (a) TE modes for various degrees of asymmetry, and (b) TM modes with asymmetry $a=1$ and $n_s/n_f=0.7, 0.76, 0.8$.

(6.10), whereas the values for cladding refractive (n_c) for both TE and TM modes of waveguides T0 and T90 were calculated with Eqs. (6.5) and (6.7), with a schematic representation shown in Fig. 6.16. While a slab waveguide similar to T0 supports the TE-mode orders $\nu=0$ and 1, it supports only the fundamental TM-mode. However, the situation is different for a waveguide similar to T90, where only the fundamental TE-mode is supported, whereas TM is cut-off.

Figure 6.16: Schematic representation of cladding refractive index n_c for TE and TM modes in slab waveguides with two different film orientations in cladding layer.

It should be noted that depending on the asymmetry value, there is an entire range of refractive indices that support a given order mode, and this is the reason why a refractive index band corresponds to each modal number in Fig. 6.16. Looking at b - V curves of Fig. 6.15, a range of b values corresponds to a given V value, thus resulting in a range of refractive indices obtained. However, knowing the large asymmetry that applies to our waveguides, the lower bound refractive index values such as $n_{TE}=1.501$ and $n_{TM}=1.545$ for T0 and $n_{TE}=1.534$ for T90 can provide enough evidence to determine the optical anisotropy of the cladding layer.

The cladding refractive index values of Fig. 6.16 for TE and TM modes of a slab waveguide similar to the waveguide T0 are quite different. For a given

waveguide, the difference

$$\Delta n_c = n_{TM} - n_{TE} \quad (6.11)$$

can characterize the birefringence properties of the cladding layer. With numerical values shown, the refractive index difference $\Delta n_c=0.044$ demonstrates a highly birefringent film. This value was found in complete agreement with experimental results of Ref. [111], where birefringence of GLAD TiO_2 films at different flux incidence angles is measured, showing a birefringence $\Delta n_c=0.05$ that corresponds to $\alpha=80^\circ$. It was also concluded that the maximum birefringence $\Delta n=0.065$ would correspond to TiO_2 films deposited at $\alpha=65^\circ$, with a critical packing density at about 0.597, [112] and it decreases with α .

The influence of characteristic packing density and column orientation on GLAD film birefringence [22] has demonstrated that the primary factor is the characteristic packing density of the columns, with column orientation playing a secondary role. In contrast, a comparison between cladding refractive indices of T0 and T90 of Fig. 6.16 reveals that the column orientation plays an important role on the waveguide modal behavior. The refractive index for TE modes in the waveguide T90 is higher than the one for the T0; this is demonstrated through the support of only the fundamental TE mode in T90. With the assumption that both films have the same packing density ($\alpha=85.0\pm0.1^\circ$), we are able to separate the influence of film structural orientation on birefringence properties. Based on this, film orientation can be considered as an alternative approach in adjusting film refractive index in general, and birefringence in particular.

The stronger influence that column orientation has demonstrated in our application could be due to a different modal interaction as compared to other works. [112] As previously pointed out, birefringent properties of GLAD films

usually are observed for light propagating through the film normal to the substrate, and the findings apply accordingly. However, this is not the case with our waveguides, where the mode interacts with the birefringent cladding and light propagates parallel to the substrate. In such case, the limited interaction of the evanescent field is compensated by the long propagating distance, or the interaction length L_{int} , resulting in a stronger influence.

6.4 MZI optical characterization

As previously mentioned, the basic structure of the MZI for sensing application consists of single mode waveguiding channels and a sensing window etched in one of the arms, where ZrO_2 or TiO_2 structured thin film was later deposited. Length of the sensing window defines interaction length of the evanescent field with the sensing environment.

Optical characterization of fabricated MZI was accomplished by measuring normalized power at the output (P_{out}/P_{in}) as a function of wavelength λ of a linearly polarized light (either TE or TM). Measurements were completed with the setup shown in Fig. 6.1. A typical wavelength response obtained for a MZI with TiO_2 slanted columns and interaction length $L=3.4$ mm is demonstrated in Fig. 6.17. High frequency noise of the wavelength response was filtered out with a low pass filter with cut-off frequency of 120 Hz and sampling frequency of 9 KHz. Filtered data are plotted on the same axis with the unfiltered ones.

The Fourier transform analysis of the filtered data gave the magnitude and phase component of each frequency. A curve fitting procedure based on the least square fitting algorithm was adopted. Initial values for magnitude and phase of dominant frequency component provided from FFT analysis were fed into fitting algorithm. A periodic function was fitted to the filtered data, yielding a transfer

Figure 6.17: A typical transmission spectra of MZI with Si_3N_4 waveguide.

function model of the MZI as shown in Fig. 6.18.

As fitting parameters, the *coefficient of determination* (R^2) which is the proportion of the variance of model/graph, and *Chi Square* value ($Chisq$) which is the sum of the squared error between the original data and the calculated curve fit, indicate how well the calculated curve fits the original data. For fitting in Fig. 6.18, $R^2=0.78$ and $Chisq=420$ were obtained. Of course, these poor fitting parameters result from the anomalous deep absorption at wavelength 1480-1560 nm. Note that outside this range a much better fitting is achieved.

The normalized output intensity of the MZI is plotted in Fig. 6.19. For comparison, the transfer function obtained in Section 3.3 from simulating a MZI model with similar characteristics at wavelength $\lambda=1550$ nm is also shown. While a good agreement is achieved at wavelength range 1540-1620 nm (where the device was simulated), there is some phase variation outside this range. The sinusoidal function, with a fixed phase/amplitude fitted to measurement data, does not take into

Figure 6.18: Fitted transfer function model and filtered data of a MZI.

account a wavelength dependent phase or modulation frequency of the MZI, thus resulting in such discrepancies.

After completing the optical characterization of a MZI device with a structured thin film deposited in one arm, we are ready to move to the next phase of demonstrating the device ability to sense the refractive index change in the sensing environment.

6.5 Refractive index measurement of CO₂

Optical measurements for gas refractive index determination were completed with the setup shown in Fig. 6.1. However, while the optical part remains unchanged, some modifications were made to include a gas flow system able to create a gas environment surrounding the sensing arm of the MZI. The gas flow system outlined in the next section was utilized to measure the refractive index of CO₂ with N₂ as

Figure 6.19: MZI transfer function measured versus simulated.

a reference.

6.5.1 Experimental setup for gas flow system

To control the gas environment surrounding the MZI with sensing window, an acrylic cell with dimensions $10 \times 15 \text{ mm}^2$ was attached to the chip surface with a double sided adhesive tape. A gas leak test completed on a dummy silicon wafer demonstrated the capability of this attachment method. Flexible tubing connected to the input hose barb on the gas cell supplied gas inside the cell. A schematic of experimental setup is shown in Fig. 6.20. Gas flow control was achieved with a three port switch, a needle valve, and a pressure regulator connected at each gas line. Gas cylinders of compressed nitrogen 99.998% purity and carbon dioxide 99.995% purity supplied the gas to the cell.

A photograph of the overall system, including optical setup and gas flow installations is shown in Fig. 6.21(a). Figure 6.21(b) is a close up view of the waveguide

Figure 6.20: Schematic of experimental setup for N_2 and CO_2 refractive index measurement.

substrate with the acrylic gas cell attached, the input tapered fiber, and the output lens. A vacuum pump connected to the holder provided a secure connection of waveguide substrate to the stage.

Figure 6.21: Photographed images of the setup for gas refractive index measurements (a) overall system with optical and gas flow components, and (b) close up view of a waveguide substrate with a gas cell attached, and input/output coupling components.

6.5.2 Temperature in the gas-cell

Experimental work started with the measurement of gas temperature inside the gas cell. A type K thermocouple probe from OMEGA Corp. of diameter 0.25 mm was inserted inside the cell through the output barb as shown in the photograph of Fig. 6.22. Thermocouple wires were connected to the hand-held unit (OMEGA HH802U), allowing temperature readings as gas flows inside the cell. Air temperature inside the cell before flowing any gas was recorded ($T=21.1^{\circ}\text{C}$). First, nitrogen was flown for 3 min inside the gas cell, with temperature reading showing fluctuation of no more than $\pm 0.1^{\circ}\text{C}$. Next, carbon dioxide was flown, temperature fluctuated for no more than -0.1°C . The experiment was repeated several times, by alternating N_2 and CO_2 at different flow time intervals, with results being consistent.

Figure 6.22: Photograph of experimental setup for temperature measurement inside gas cell. A type "K" TC probe connected to the hand held unit for temperature reading was inserted through the gas output barb into the gas cell.

Having defined a $\Delta T = \pm 0.1^\circ\text{C}$, a quick analysis on temperature-induced change in the index of refraction for ZrO_2 and TiO_2 films can be completed. The refractive index of TiO_2 film with thermo-optic coefficient [113]

$$\frac{\Delta n}{\Delta T} = (-1.77 \pm 0.7)10^{-4}[K^{-1}] \quad (6.12)$$

is expected to change by a $\Delta n = 3.54 \times 10^{-5}$ for a temperature variation of up to 0.2°C in the gas cell. This change in the refractive index will cause a change in the effective index of the propagating mode $\Delta N_{eff} = 2.7 \times 10^{-6}$, corresponding to a phase shift $\Delta\phi = 0.01\pi$ in the MZI output spectrum.

Similar calculations were completed for ZrO_2 film with thermo-optic coefficient [114]

$$\frac{\Delta n}{\Delta T} = (3 \pm 0.5)10^{-5}[K^{-1}] \quad (6.13)$$

For $\Delta T = \pm 0.1^\circ\text{C}$, calculated values of temperature induced phase shift were found below 0.01π . Therefore, the maximum shift in the interferometer output spectrum induced by the thermal variations in the measuring environment is significantly lower than the shifts predicted by simulation results, demonstrating that gas temperature fluctuations as measured will not obstruct optical measurements; they can be neglected and a temperature controlled sample holder will not be required in the measurement process.

6.5.3 Measurement of gas refractive index

As gas flows into the cell, the environment surrounding the sensitive layer changes accordingly, resulting in a refractive index change within the evanescent field region. With relation of Eq. (1.5), this refractive index change affects the effective index and hence, induces a phase shift $\Delta\phi$ in the MZI transfer function. When the

transfer function of a MZI is plotted as a function of the wavelength [$P_{out}=f(\lambda)$], this phase change will shift the response accordingly, reflecting any change in the sensing environment. Measuring this $\Delta\phi$ after introducing a new gas into the cell gives the ability to determine Δn and hence the gas refractive index.

Measuring process started on devices with ZrO_2 structured thin film deposited in the sensing window. The gas content into the cell was alternated from N_2 to CO_2 in 5 min intervals and transmittance for TE and TM polarized light was measured at each interval. Measurement data, together with corresponding fitted transfer function models, are shown in Figures 6.23(a) and 6.24(a). A reasonable fitting quality was provided, with correlation coefficient values in the range 0.92-0.93.

Based on the transfer function models, normalized output intensity that corresponds to N_2 and CO_2 into the gas cell is plotted in Figs. 6.23(b) and 6.24(b). From these plots, phase shift, as result of gas content change in the sensing environment, can be measured. To demonstrate, device output intensity in Fig. 6.23(b) is shifted by $\Delta\phi=\pi/16$ when CO_2 is introduced into the sensing environment. In addition, change in output intensity at a given wavelength can be extracted from the plot. The correspondence between the intensity value and gas refractive index at $\lambda=1620$ nm, is shown in Fig. 6.25. The error bars present one standard deviation of the intensity value obtained after analyzing data sets from three repeated measurements. Similarly, phase shift and the intensity values corresponding to TM polarized light were measured and are shown in the same axes. Similarity in slope proves that both polarizations provide comparable intensity changes and will work well in a sensing application.

Further, with Eq. (2.1) and the phase shift $\Delta\phi=\pi/16$ obtained from Fig. 6.23(b), the change in effective refractive index and the device sensitivity

Figure 6.23: MZI with ZrO_2 film and N_2/CO_2 in sensing area with TE polarized light (a) transmittance and fitted transfer function, and (b) normalized intensity.

Figure 6.24: MZI with ZrO_2 film and N_2/CO_2 in sensing area with TM polarized light (a) transmittance and fitted transfer function, and (b) normalized intensity.

Figure 6.25: Output intensity change at $\lambda=1620$ nm for TE and TM polarization when gas is switched from N_2 to CO_2 .

were calculated:

$$\Delta N_{eff} = \frac{\Delta\phi\lambda}{2\pi L} = 7.6705e - 6; \quad (6.14)$$

$$S = \frac{\Delta N_{eff}}{\Delta n} = 0.0489 \quad (6.15)$$

Measuring process continued on devices with TiO_2 structured thin film deposited in the sensing window. The gas content into the cell was alternated from N_2 to CO_2 in 5 min intervals and transmittance for TE and TM polarized light was measured at each interval. Measurement data together with corresponding fitted transfer function models are shown in Figs. 6.26(a) and 6.27(a). A good fitting quality was provided, with correlation coefficient values in the range 0.95-0.97.

Based on the transfer function models, normalized output intensity corresponding to N_2 and/or CO_2 into the gas cell is plotted in Figs. 6.26(b) and 6.27(b).

(a)

(b)

Figure 6.26: MZI with TiO_2 film and N_2/CO_2 in sensing area with TE polarized light (a) transmittance and fitted transfer function, and (b) normalized intensity.

(a)

(b)

Figure 6.27: MZI with TiO_2 film and N_2/CO_2 in sensing area with TM polarized light (a) transmittance and fitted transfer function, and (b) normalized intensity.

From these plots, phase shift as a result of gas content change in sensing environment can be measured. To demonstrate, device output intensity in Fig. 6.26(b) is shifted by $\Delta\phi=\pi/10$ when CO_2 is introduced into the sensing environment. In addition, change in output intensity at a given wavelength can be extracted from the plot. The correspondence between intensity value and gas refractive index at $\lambda=1620$ nm is shown in Fig. 6.28. Similarly, phase shift and the intensity values corresponding to TM polarized light were measured and are shown in the same axes. Similarity in slope proves that both polarizations provide comparable intensity changes and will work well in a sensing application. It should be noted here the negative slope obtained for TM polarization. In general, slope sign depends on where the device operation point is located and the slope of the fringe on that location.

Figure 6.28: Output intensity change at $\lambda=1620$ nm for TE and TM polarization when gas is switched from N_2 to CO_2 .

With Eq. (2.1) and the phase shift $\Delta\phi$ obtained from Fig. 6.26(b), change in

effective refractive index and the device sensitivity were calculated:

$$\Delta N_{eff} = \frac{\Delta \phi \lambda}{2\pi L} = 1.8837e-5; \quad (6.16)$$

$$S = \frac{\Delta N_{eff}}{\Delta n} = 0.1200 \quad (6.17)$$

Using a point-slope formula of form $y - y_0 = m(x - x_0)$, device sensitivities for the MZI with ZrO_2 and TiO_2 films deposited on the sensing window are shown in Fig. 6.29. The error bars that correspond to experimental results present one standard deviation of the ΔN_{eff} calculated after analyzing data sets from three repeated measurements. For comparison, simulated sensitivity is also plotted, showing a good agreement. As expected, higher sensitivity has been achieved by the device with TiO_2 film. The main factor contributing on higher sensitivity is the refractive index of TiO_2 cladding which is closer to the refractive index of the Si_3N_4 core.

Figure 6.29: Sensitivity comparison for devices with ZrO_2 and TiO_2 film.

Such behavior was predicted by the simulated sensitivity curve shown in Fig. 2.1, where a higher refractive index of sensing layer shifts the device operation point toward the higher slope region, thus providing better sensing abilities. Results shown in Fig. 6.29 suggest that sensitivity is strongly affected by the core-cladding refractive index difference. In general, this demonstrates the ability of the optical sensing platform where a sensor can be fabricated by combining a dielectric structured thin film with a planar waveguide, and a good refractive index matching will enhance device sensitivity.

6.5.4 Time response

Time response of a MZI can also be employed for refractive index determination. With a similar setup and operating conditions as described in the previous section, the output time response was measured for alternating N_2 and CO_2 gas flow on the sensing environment. Measurements were completed for TE polarized light at $\lambda=1620$ nm. This wavelength provides a device point of operation well-off the sensitivity fading region, where the slope of the fringe pattern becomes zero. As a result, device will operate on the linear part of the transfer function, where any change in the refractive index value would correspond to a change in power output.

Gas flow in the cell was alternated from N_2 to CO_2 with the corresponding output signal shown in Fig. 6.30. Note that the frequency noise coupled with the output signal was filtered out before plotting it. From Fig. 6.30(a), for a device with ZrO_2 film, the output signal changes by $\Delta P_{out}=0.38$ dBm when gas in the sensing environment changes from N_2 to CO_2 . This change agrees with the ΔP at $\lambda=1620$ nm obtained in Fig. 6.23(a). A similar behavior can be seen in Fig. 6.30(b), which shows output power with time for a device with TiO_2 film.

The output signal drops by $\Delta P_{out} = -0.65$ dBm when CO₂ is introduced in the gas cell. Note that the sign in ΔP_{out} depends on the wavelength; in other words in operation point location on the intensity fringe curve.

While the output time response patterns look similar in both devices, there is a quantitative difference. The device with TiO₂ film in the sensing layer demonstrates a larger $|\Delta P_{out}|$, which agrees with what was previously concluded; that devices with TiO₂ film offer higher sensitivity. Also, looking at time response plots, a qualitative observation can be drawn. While the output power changes instantly when switching from N₂ to CO₂, there is a time constant when switching from CO₂ to N₂; it takes approximately 200 s to fill the 1.2 μm thick NTF with CO₂ but only 80 s to replace it with N₂. This could be due to a higher density of the CO₂ as compared to N₂, or due to a higher sticking coefficient of CO₂, a parameter used in surface physics to describe the ratio of the number of molecules that do adsorb to a surface to the the total number of atoms that impinge upon that surface during the same period of time. As a result, CO₂ molecules would adsorb to oxide to a greater degree than N₂. Thus, both these factors may contribute in introducing a time constant required for CO₂ to be removed from sensing environment.

6.5.5 Sources of high frequency noise on the output signal

Gas flowing inside the cell and in the film itself disturbs columns and their interaction with the EM field propagating in the vicinity. It is related mainly with mechanical properties of the film where the aspect ratio diameter/length of the columns is in the order of 1/14. Experimental work has shown that the films undergo elastic deformation when small forces are applied to the surface, [19] corresponding to stiffness as low as 1/1000th that of similarly deposited solid film.

Figure 6.30: Time response for alternating N_2 and CO_2 in the gas cell on device with (a) ZrO_2 , and (b) TiO_2 film

Deflection of slanted posts considered as cantilevers with rectangular cross-section can be calculated with a theoretical expression, [115]

$$\Delta x = \frac{FL^3 \cos^2(\alpha)}{3EI} \quad (6.18)$$

where F is the applied force, L is cantilever length, α is the rise angle, E is Young's modulus, and I is the moment of inertia (for rectangular cross section $I = wt^3/12$, where w is the width and t is the thickness). [115] Depending on the applied force, this displacement can be destructive or nondestructive. As calculated for a SiO_2 film, [115] a nondestructive displacement might exhibit resonant frequency in the range 10-50 MHz.

To evaluate the displacement contribution on high frequency noise coupled with our output signal, a quick calculation with Eq. (6.18) was completed. For $F=5e-9$ N [116], $L=1.26$ μm , $\alpha=45^\circ$, $E=120e9$ Pa, $w=120$ nm, and $t=120$ nm, the displacement was found to be $\Delta x=7.6$ nm. This displacement is comparable with results obtained in literature for similar films; it causes high frequency noise. This evaluation was supported by observations during experiments where noise amplitude was increased with gas flow rate.

Gas temperature would be another factor contributing to high frequency noise. As was pointed out previously, temperature fluctuations due to the gas flow in the cell will not cause significant shift in the output signal; however, when the signal is weak and signal to noise ratio becomes small, they will introduce some noise. It is believed that thermo-optic noise, together with the mechanical disturbances in the film columns, affected the output signal, especially the time response.

6.6 Summary

The optical measurements and characterization presented in this chapter concluded the overall simulation and fabrication work of the project, by validating the newly proposed platform for interferometric-based sensing applications. In addition, the optical loss analysis of fabricated waveguides, with and without structured thin film deposited on the sensing area, were presented.

Based on measurement results, an optical loss analysis on simple waveguides with SiO₂ over-cladding layer of thickness 1.8 μm was completed. With the application of cutback technique, waveguide attenuation coefficient at $\lambda=1.55 \mu\text{m}$ was obtained. High absorption losses with attenuation coefficient $\alpha=7.1 \text{ dB/cm}$ at telecom wavelength were found in agreement with other works. [106] Such high waveguide attenuation was attributed to the excessive amount of hydrogen introduced into the Si₃N₄ film during the deposition process. While an attempt was made to reduce the hydrogen content through an annealing procedure, it showed no improvement on the waveguide attenuation, demonstrating that annealing process at 600°C was not effective. The hydrogen still remained trapped in the waveguide structure.

While the high waveguide attenuation at $\lambda=1.48\text{-}1.56 \mu\text{m}$ was due to the intrinsic material absorption, waveguides perform much better outside this range. As the wavelength approaches C and L bands, absorption and scattering losses were reduced substantially, reaching a value of 0.62 dB/cm at $\lambda=1.62 \mu\text{m}$, thus demonstrating high quality of fabricated waveguides, with low intrinsic absorption and scattering due to inhomogeneity and surface roughness. The fabrication process employed on the samples achieved smooth core-cladding interface. Absorption losses due to high hydrogen content on the Si₃N₄ layer can be overcome by operating the device at wavelength $\lambda=1.62 \mu\text{m}$.

The analysis continued with segmented waveguides where SiO_2 over-cladding was removed in an area $24 \times 3100 \mu\text{m}^2$ above the ridge and replaced by ZrO_2 or TiO_2 structured thin film of different configurations. Measurement results showed no correlation between structured thin film material deposited in the segment and the waveguide losses. However, several trends were found showing that (a) TE polarized light propagates with lower losses than the TM; (b) samples with structured thin films of slanted columns have smaller losses, as compared to samples with vertical columns; and (c) samples with structured films of slanted columns, but different orientations, showed similar losses.

It is of interest to point out that some anomalous behavior was observed for waveguides with TiO_2 films. The highly birefringent properties of TiO_2 , deposited with the GLAD technique, affected the device output optical power. A more detailed analysis, completed on a slab waveguide with the applied refractive indices and waveguide geometry, demonstrated that the device with film of slanted columns tilted in the propagation direction supports two modes for TE polarized light and one mode for TM polarization, thus causing a wavelength dependent output. On the contrary, a device with columnar film tilted perpendicular to the propagation direction supported only one TE mode and cuts-off the TM mode. Based on the modal analysis completed on two typical samples, it was concluded that the refractive index for TE and TM mode is different, indicating birefringent properties of the film. A refractive index difference $\Delta n = 0.044$ obtained with this analysis was found in complete agreement with experimental works [111] published for similar films.

With the assumption that films on both measured samples have the same packing density, we were able to separate the influence of film structural orientation on birefringence properties. Quite surprisingly, a comparison between cladding

refractive indices of considered waveguides revealed that the column orientation plays an important role on the waveguide modal behavior. The stronger influence that the column orientation has demonstrated in our applications could be due to the modal interaction of light propagating in a direction parallel to the substrate as opposed to the commonly seen cases where light propagates through the film normal to the substrate. The limited interaction of the evanescent field with the birefringent cladding is compensated by the long propagating distance.

Finally, the optical measurements on interferometer-based devices with ZrO_2 or TiO_2 film deposited on the sensing area were completed. Results obtained demonstrated the optical sensing capability of such device through the refractive index measurement of a known gas. A gas flow system setup capable of supplying N_2 or CO_2 was adopted. By alternating gas content in the sensing environment, a maximum phase shift of $\Delta\phi=\pi/10$ on the MZI transmission spectra was achieved. Devices with TiO_2 film deposited on the sensing area performed much better than devices with ZrO_2 , with a sensitivity twice as high. In addition, measured response was found in good agreement with simulation results.

Sensing ability was confirmed by measuring time response at the device output as well. While the output time response pattern looks similar in both devices, there is a quantitative difference. Device with TiO_2 film in the sensing layer demonstrated larger $|\Delta P_{out}|$, which agrees with what was previously concluded; that devices with TiO_2 film offer higher sensitivity. Also, looking at time response plots, a qualitative observation can be drawn. While output power changes instantly when switching from N_2 to CO_2 , there is a time constant when switching from CO_2 to N_2 . This can be explained with the CO_2 being heavier than N_2 ; some time would be required for CO_2 to be removed from sensing environment.

In addition, a brief analysis on high frequency noise associated with the measured response has shown that gas temperature fluctuation as well as the elastic deformations of the columnar structure could be the source.

Chapter 7

Conclusions and Future Work

In this thesis, a new platform for interferometry-based optical sensing devices based on the application of nanostructured dielectric thin films as a sensitive environment was developed. A large surface area per volume and the ability to draw the evanescent field deep into the cladding, increased the optical-mode measurand interaction, making these films very attractive for optical sensing.

7.1 Summary and conclusions

Integrated optical sensing in general, and interferometry-based devices in particular, were found to be an excellent medium to demonstrate the application of a new optical sensing platform. In addition, the potential to combine the optical sensing devices with a multi-function electronic micro-system integrated in the same chip, makes such a medium very appealing for a large range of applications.

After building a short theoretical foundation for interferometric-based optical sensing devices, the waveguide technology, the technique for sensitive layer deposition, and the appropriate dielectric materials for optical sensing applications were obtained. In regard to the waveguide technology, Si_3N_4 was found to offer several advantages. In addition to compatibility with IC technology and low

optical losses, its refractive index can be broadly adjusted during the fabrication process, thus allowing a wide range of dielectric films to be deposited on the cladding layer. Given that the GLAD technique can provide nanostructured thin films with a very large ratio of surface area to volume, deposition of dielectric porous films of Al_2O_3 , ZrO_2 and TiO_2 with this technique, was found suitable for our application. In a comparison to other methods of creating porous films, the GLAD alternative appears advantageous. With just one deposition step, this technique provides remarkable possibilities to tailor the film structure to a specific application. Further, the dielectric materials selected for this application provide refractive index variation ranging from 1.50 to 2.22, permitting some flexibility on layer porosity, without compromising waveguide losses. Their low absorption and scattering losses, and good adhesion to Si_3N_4 core layer were also considered in the selection process.

Simulation and modeling work evaluated the interferometric sensing ability of a Mach-Zehnder interferometer device with structured thin film deposited on the sensing area. Initially, a single-mode Si_3N_4 waveguide was designed. This was followed by modeling and simulation of dielectric structured thin film growth. Different flux incidence angles during the growth process resulted in films with very different morphologies and porosities. The refractive index value of each film was estimated based on film porosity and bulk material refractive index. In addition to micro-structural growth orientation, simulation results predicted that depending on the incidence angle during growth process, a 20-30% variation in the film refractive index can be achieved. The analyzed growth parameters, such as flux incidence angle and angular velocity of a rotating substrate, demonstrated a quite significant effect on film refractive index values. These parameters were crucial in modeling and simulation of segmented waveguide and Mach-Zehnder

interferometer-based evanescent field sensors with structured thin film deposited on the sensing area. Simulation of such device was completed for two different values of refractive index of the pore content, corresponding to N_2 and CO_2 . As simulation results predicted, such refractive index change would induce a 10 nm shift in the optical transmission spectra of the device, resulting in a 7% change in output intensity.

In the fabrication process, a new method for Si_3N_4 low temperature plasma etching was established. Compatibility with integrated circuit process and the application of a carbon free etching based on SF_6/O_2 fluorine chemistry mixture in ECR plasma source makes this method attractive to a wide range of applications such as optical and MEMS devices. As demonstrated, the combination of the fluorine chemistry mixture in ECR plasma source, with a low substrate temperatures at about $-30^\circ C$, can successfully etch a $0.4\ \mu m$ deep Si_3N_4 channel. This newly developed process was adopted to fabricate planar waveguides and optical devices in eight almost identical prototype wafers. Wet etching of patterned wafers allowed SiO_2 cladding to be removed from sensing areas, thus creating the environment for final deposition of structured thin film.

In the early stages of fabrication process, dielectric structured thin films of Al_2O_3 were deposited on stationary glass substrates. With a maximum flux incidence angle of $\alpha=73^\circ$, these films demonstrated columnar growth, however with a limited porosity. Measurement data collected from these samples were essential on defining parameters for the second round of structured thin film deposition. Dielectric films of ZrO_2 and TiO_2 were deposited on both stationary and rotating processed wafers. As a result, eight different material-nanostructure combinations were obtained, including slanted and vertical columnar structures. This relatively large quantity of samples permitted the analysis of the effect of film material and

nanostructure orientation on device modal behavior and sensing ability.

Optical characterization of Al_2O_3 structured thin films deposited on stationary, tilted glass substrates consisted in refractive index determination as well as birefringence property investigation. Refractive index determination was approached with the application of the ellipsometry measurements and a single-layer optical model. Films deposited at different flux incidence angles were measured. As predicted by the simulation results, these measurements demonstrated a strong relation between flux incidence angle, film porosity and its refractive index. Films deposited at $\alpha=71^\circ$ have a higher refractive index than films deposited at $\alpha=73^\circ$, where a $\Delta\alpha=2^\circ$ was able to produce a change in refractive index $\Delta n=0.06$.

Film birefringence investigation was completed by measuring wavelength and orientation dependent transmittance of light propagating normal to the substrate plane. By introducing an Al_2O_3 coated glass substrate in between two crossed polarized lenses, the output power at the detector ($\lambda=450$ nm) was increased to 14% of the maximum power corresponding to lenses in $\mathcal{P}$ -state. Orientation dependence transmittance was also observed; a film layer of thickness 840 nm achieved a 4° polarization rotation angle with the optical power at the output oscillating from 0 to 11 μW . Further, by changing the azimuthal angle to $\Phi=70^\circ$, the output power increased to 130 μW . These results demonstrate a large film birefringence which might be beneficial to a different application such as polarizing components, waveplates, and retarders.

Based on measurement results, an optical loss analysis on fabricated linear waveguides was completed. High absorption losses of Si_3N_4 waveguides at telecom wavelength were found in agreement with other sources. [106] However, as wavelength approaches C and L bands, absorption and scattering losses were reduced substantially, reaching the value of 0.62 dB/cm at $\lambda=1.62$ μm . Optical

losses on segmented waveguides with ZrO_2 and TiO_2 films deposited on a section of length 3-4 mm were also completed. Measurement results showed no correlation between film material deposited in the segment and the waveguide losses. However, several trends were found showing that (a) TE polarized light propagates with lower losses than the TM one; (b) samples with structured thin films of slanted columns have smaller losses as compared to samples with structured film of vertical columns; and (c) samples with structured films of slanted columns but different orientations showed similar losses. The highly birefringent properties of TiO_2 deposited with the GLAD technique affected the device output optical power. A more detailed analysis completed on a slab waveguide with the applied refractive indices demonstrated that the device supports two modes for TE polarized light and one mode for TM polarization, thus causing a wavelength dependent output. Based on these analysis, refractive index for each TE and TM mode was calculated, revealing a $\Delta n=0.044$. This value was found in complete agreement with results published [111] for similar films.

The overall work of this project including design, fabrication, and characterization of these optical devices concluded with the optical measurements completed on interferometer-based device with ZrO_2 or TiO_2 film deposited on the sensing area. Results obtained demonstrated the optical sensing capability of such a device through the refractive index measurement of a known gas. By alternating gas content in the sensing environment, a maximum phase shift of $\Delta\phi=\pi/10$ on the MZI transmission spectra was achieved. Sensing ability was confirmed by measuring both frequency and time response at the device output. Devices with TiO_2 film deposited on the sensing area performed much better than devices with ZrO_2 , with a sensitivity twice as high. In addition, simulation results were found in good agreement with measurement results. A brief analysis on high frequency

noise associated with the measured response has shown that gas temperature fluctuation, as well as the elastic deformations of the columnar structure, could be the source.

7.2 Future work

As presented, this project was focused mainly on the development of a new platform for interferometric-based optical sensing with structured thin films deposited with the GLAD technique. By demonstrating optical sensing capabilities, it is our belief that we have established the groundwork for further development of more application specific devices. Of course, more design, experimental, and characterization work would be required in order to make these devices commercially available. From this perspective, there are several directions that may potentially improve device performance as well as provide new opportunities for this application. For example, a new conceptual device based on a multi mode interference coupler was demonstrated in Ref. [5].

Modeling and simulation of EM field propagating in the structured thin film layer is important for this application. The modeling software 3D-FILM was successfully employed for film growth and density evaluation. However, as pointed out in Section 3, due to lack of commercially available software, it might be beneficial to further increase the modeling capability of this software by including an EM solver, making it able to predict absorption and scattering losses of light propagating in the medium.

The experimental work in Sections 5.2.1 and 5.2.2 was focused on exploring some potential applications of nanostructured thin films on transparent substrates.

The promising results obtained demonstrate that this quite inexpensive fabrication process, may be a very attractive alternative to other methods of manufacturing wave-plates and retarders. However, more characterization work to define the effect of film thickness and density on the angle of anisotropy would bring the application closer to commercialization.

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Appendix A

Starting Wafer Parameters

Sample		C3	C6	C7	C8	C10	C11	C12	C13	C14
ID		154W	168W	168W	168W	168W	186W	186W	186W	186W
SiO ₂ base	t (nm)	3516	3509	3509	3508	3510	3509	3508	3507	3508
Si ₃ N ₄	t (nm)	1023	1022	905.5	990	1022	1013	1013	1036	1033
	A_n	1.94	1.922	1.922	1.920	1.922	1.926	1.931	1.930	1.926
	$B_n \times 10^{-3}$	7.37	9.557	9.050	7.980	9.407	6.649	5.016	6.090	7.270
	$C_n \times 10^{-4}$	7.67	5.345	4.669	6.777	4.927	6.929	9.777	7.976	6.574
	n ($\lambda=650$ nm)	1.96	1.947	1.946	1.943	1.947	1.946	1.948	1.949	1.948
	n ($\lambda=1551$ nm)	1.94	1.926	1.926	1.923	1.926	1.929	1.933	1.932	1.947
SiO ₂ cap	t (nm)	99.1	97.4	94.3	91.4	93.0	104	99	101	99
	A_n	1.495	1.501	1.493	1.496	1.507	1.475	1.463	1.469	1.470
	$B_n \times 10^{-2}$	-3.246	-3.021	-3.983	-3.652	-4.279	-2.074	-1.507	-1.576	-2.358
	$C_n \times 10^{-3}$	2.431	3.881	3.177	2.554	3.241	1.289	0.373	0.588	1.502
	n ($\lambda=650$ nm)	1.431	1.403	1.415	1.427	1.429	1.439	1.423	1.436	1.437
	n ($\lambda=1551$ nm)	1.482	1.486	1.481	1.475	1.489	1.465	1.458	1.463	1.460

Table A.1: Measurements on starting wavers, referring to stack measurements completed at NRC.

Appendix B

Device Process Flow

All processing steps with corresponding key parameters required to fabricate optical devices presented in this project are shown. In addition to the information given in Chapter 4, these detailed list would make it possible to reproduce samples for possible future applications.

- PS#1: Sample cleaning and preparation before lithography
 - Rinse wafer in DI water for 10 min
 - Bake in 200°C for 20 min
- PS#2: Photolithography and patterning
 - Spin/spread layer of HMDS (PR adhesive) at 4000 rpm for 30 sec
 - Bake on hot plate at 105°C for 1 min
 - Flood with PR S1811 and spin at 4000 rpm for 30 sec
 - Bake on hot plate at 105°C for 1min
 - Expose for 26 sec on UV radiation
 - Develop with MF-321 developer for 1 min
 - Rinse in DI water for 30 sec

- Hard bake on hot plate at 125°C for 3 min
- PS#3: Descum with O₂ plasma
 - Use Plasma Planar Etcher (Technics)
 - Base pressure 0.028 Torr
 - Precondition with O₂ for 1 min
 - Descum for 2 min with power adjusted at 100 W
- PS#4: Etch SiO₂ hard mask layer of thickness 100 nm with RIE
 - Gas mixture CHF₃/O₂ 42.5/7.5 SCCM
 - Base pressure 2.5e-5 Torr
 - RF peak 125 W
 - RF forward power 350 W
 - Etch pressure 150 mTorr
 - RF reverse 0 W
 - RF load 9.4
 - Etch for ~130 sec with expected etch rate 600 Å/min
- PS#5: Etch Si₃N₄ rib in ECR
 - Use ECR etcher 13.56 kHz
 - RF P1 power 10 W
 - RF P1 reflected power 0 W
 - RF P2 power ~300 W
 - RF P2 reflected power ~3 W

- Gas mixture SF_6/O_2 with rates 3.65/2.21 SCCM
- Wafer temperature -30°C
- Base pressure $5e-6$ (without gas flowing in)
- Operating pressure 6 mTorr
- Etch time 110 sec
- PS#6: PR stripping with microwave oven
 - Use microwave oven with O_2 gas
 - Pressure 5 Torr
 - Flow O_2 at 300 SCCM
 - Etch time 6+2 min
- PS#7: RCA pre-furnace cleaning with standard process on all wafers before LoTox deposition
 - $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ with mixing ratio 0.25:1:5 at 80°C for 5 min
 - Rinse for 10 min
 - $\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ with mixing ratio 1:1:6 at 80°C for 5 min
 - Rinse for 10 min
 - HF of concentration 1% for 30 sec
 - Rinse for 10 min
 - Spin dry
- PS#8: Deposit LoTox using furnace
 - Deposition temperature 410°C

- Base pressure 0.01 mTorr
- Rate of rise 0.03 mTorr/min
- Deposition time 90 min
- Estimated deposition rate 200 Å/min
- PS#9: Wet etching of LoTox with Siloxide etchant
 - Etch for 13-14 min with estimated etch rate 1500Å/min
 - Rinse wafer for 10 min after etching

Appendix C

Etching time and thickness measurements on processed samples

Note: wafer C6 has not gone through window patterning and wet etching of SiO_2 layer. It was designated as a reference sample.

Wafer	Mask R0109709	Etching time		SiO ₂ dep. thickness (μ m)	Mask CU27901A	SiO ₂ wet etching (min)	Rib thickness (nm)
		RIE (sec)	ECR (sec)				
3	Y	130	110	N/A	Y	14	125 $\pm$ 7
6*	Y	140	120	60	N/A	N/A	N/A
7	Y	140	120	60	Y	13	131 $\pm$ 3
8	Y	140	120	60	Y	13	120 $\pm$ 3
10	Y	130	110	N/A	Y	14	127 $\pm$ 2
11	Y	130	110	N/A	Y	14	126 $\pm$ 7
12	Y	130	110	N/A	Y	14	105 $\pm$ 13
13	Y	130	119	N/A	Y	14	112 $\pm$ 4
14	Y	130	110	N/A	Y	14	129 $\pm$ 9

Table C.1: Processing steps completed on nine wafers, thickness of deposited SiO₂ overladding and rib thickness of finished samples.