

SILICON OXYNITRIDE LAYERS FOR APPLICATIONS IN OPTICAL WAVEGUIDES

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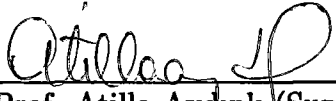
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FİZİK BÖLÜMÜ MERKEZİ**

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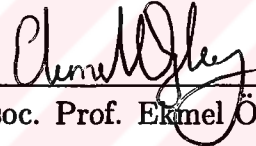
Feridun Ay

September 2000

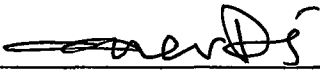
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Abstract

SILICON OXYNITRIDE LAYERS FOR APPLICATIONS IN OPTICAL WAVEGUIDES

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Silicon oxynitride layers, aimed to serve as the core material for optical waveguides operating at $1.55\mu\text{m}$, were grown by a PECVD technique using SiH_4 , N_2O , and NH_3 as precursor gases. The films were deposited at 350°C , 13.56 MHz RF frequency, and 1 Torr pressure by varying the flow rates of N_2O and NH_3 gases. The resulting refractive indices of the layers varied between 1.47 and 2.0.

The compositional properties of the layers were analyzed by FTIR and ATR infrared spectroscopy techniques. A special attention was given to the N-H bond stretching absorption at $3300\text{-}3400\text{ cm}^{-1}$, since its first overtone is known to be the main cause of the optical absorption at $1.55\mu\text{m}$. An annealing study was performed in order to reduce or eliminate this bonding type. For the annealed samples the corresponding concentration was strongly reduced as verified by FTIR transmittance and ATR methods.

A correlation between the N-H concentration and absorption loss was verified for silicon oxynitride slab waveguides. Moreover, a single mode waveguide with

silicon oxynitride core layer was fabricated. Its absorption and insertion loss values were determined by butt-coupling method, resulting in low loss waveguides.



Keywords: PECVD, Silicon oxynitride, FTIR, ATR, Waveguide, Optical absorption, Single mode, Prism coupling, Annealing.

Özet

OPTİK DALGA KILAVUZLARI UYGULAMALARI İÇİN SİLİSYUM OKSİNİTRAT TABAKALAR

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Silisyum oksinitrat tabakalar, $1.55\mu\text{m}$ optik dalgaboyunda çalışan dalga kılavuzlarının çekirdek kısmını oluşturması amaçlanarak, SiH_4 , N_2O , ve NH_3 gazları kullanılarak PECVD tekniği ile büyütüldü. Tabakalar 350°C 'de N_2O ve NH_3 gazlarının akış oranları değiştirilerek elde edildi. Büyütülen tabakaların kırılma indisleri 1.46 ile 2.00 arasında değiştirilebilmektedir.

Büyütülen tabakalar FTIR ve ATR yöntemleri ile incelendi. 3500 cm^{-1} deki N-H salınımının overtonunun $1.55\mu\text{m}$ de soğurmaya neden olduğu bilindiğinden bu bant ayrıntılı incelendi. Isıl tavlamanın tabakaların yapı özelliklerine etkileri araştırıldı. FTIR ve ATR yöntemleri ile tavlama sonucu N-H salınımının tamamen ortadan kaldırıldığı gösterildi.

Silisyum oksinitrata dayalı düzlemsel dalga kılavuzları üretildi ve optik soğurum N-H bağ sayısı ile ilişkilendirildi. Ayrıca, SiON 'a dayalı tek kipli dalga kılavuzları da üretilerek bunlardaki giriş kaybı ile optik kayıp ölçüldü. Sonuç olarak düşük kayıplı dalga kılavuzları üretildi.

Anahtar

sözcükler: PECVD, Silisyum oksinitrat, FTIR, ATR, Dalga kılavuzu, Optical kayıp, Tavlama, Tek kip şartı, Prisma çiftlemesi.



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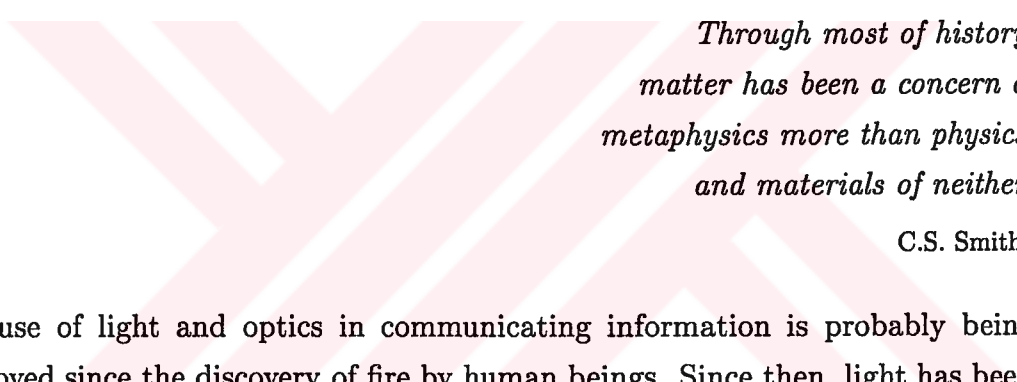
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Chapter 1

Introduction



*Through most of history,
matter has been a concern of
metaphysics more than physics,
and materials of neither.*

C.S. Smith¹

The use of light and optics in communicating information is probably being employed since the discovery of fire by human beings. Since then, light has been used to exchange important military and everyday life information both on the sea and on the ground. Today, information affects every aspect of our lives because of the enormous improvements in the computer and communication technologies.² Communication industry has become a leading force in the world's economy. Everyone would agree that our lives would be totally different without computers, cell-phones, internet, satellites, and many other information processing tools. The increasing importance of information access and manipulation, forces us to process it faster, with fewer inaccuracies, at a lower cost, and with a system which consumes less space.

1.1 Integrated Optics

The basic design of optical systems has not changed for hundreds of years. They had bulky and heavy components requiring careful alignment, protection against vibration, moisture, and temperature drift. In the early 1970's, in order to make them more compatible with modern technology, integrated optics concept emerged.³ At that time, the availability of low loss optical fibers together with the invention of the laser caused an increasing interest in compact optical systems in which, the conventional electrical integrated circuits are replaced by miniaturized optical integrated circuits (OIC) and the wires and radio links are replaced by optical fibers.

Optical integrated circuits would have a number of advantages compared to the electrical integrated circuits: improved reliability, immunity to vibration and electromagnetic interference, low loss transmission, small size, light weight, large bandwidth (multiplexing capability), lower power consumption, and batch fabrication economy. Putting it in another way, the main usefulness of the OIC is obtained from utilization of the wave characteristics of optical signals.⁴⁻⁶

Initially, the formation of OIC was considered to be achieved by simply extending the technology of microelectronics. However, optical wavelengths are of the order of 10^6 times smaller than the radio waves, which lead to a conclusion that new materials and new methods are needed. Therefore, the research in integrated optics has concentrated around two primary goals. One is to apply thin film technology to construct optical devices and circuits. They include lasers, detectors, switches, modulators, prisms, lenses, and polarizers, which have better efficiencies than their bulk counterparts. The other is the integration of a large number of optical devices, consisting of three basic components: source, waveguide, and detector, on a small substrate.

Depending on the process of integration and on the materials used, OIC's are classified in two basic types.⁷ One of them is the monolithic optical integrated circuit, in which a single substrate material is used for all devices. Because of the requirement of a source of light, monolithic circuits can only be

fabricated in optically active materials, such as direct band-gap III-V compound semiconductors in the periodic table. The other is the hybrid OIC, in which the performances of different devices are optimized by bonding together two or more substrate materials. Here the source, the waveguide, and the detector are made of compound semiconductor and dielectric materials.

Although the monolithic-type OIC seems to be ideal for optical integrated circuits its implementation proves to be rather difficult. The reason is that semiconductors' higher transmission loss and lower electro-optic coefficient makes them less effective for guiding and modulation purposes. On the other hand, the hybrid-type OIC is relatively easy to fabricate, but it has a problem in assembling the components together. However, it is still more advantageous since the appropriate materials and processing techniques can be easily utilized.

1.2 Motivation and Advantages of Silicon Oxynitride Technology

Since the introduction of integrated optics notion in 1970's, extensive studies have been carried out on a variety of optical materials such as III-V semiconductors, lithium niobate, polymers, and silicon based compounds.⁸

III-V Semiconductors

Work on III-V semiconductors was motivated mainly by the possibility of construction of monolithic OICs. Material systems such as *GaAs/AlGaAs* and *InGaAsP/InP* were used for construction of basic devices.^{4,9-12} A considerable amount of research resulted in fabrication of required components including waveguides (unarguably the basic component of photonic integrated circuits), detectors, modulators, and lasers. The optical waveguides, besides connecting other devices, were also incorporated in many functional waveguide devices. Fabrication of waveguides requires that the refractive index of the guiding region be greater than the index of its surround. The particular usefulness of the III-V

(or II-VI) ternary and quaternary compounds comes from the fact that they are optically active and that their energy band gap can be changed over a wide range by altering the relative concentration of elements. Especially *AlGaAs* technology has been well developed compared to other III-V ternaries. Its unique property is that the lattice constants of *GaAs* and *AlAs* are almost identical (5.646 and 5.396 Å respectively), which comes to be useful in fabrication of multilayered devices. Another important output of lattice matching technology is *GaInAsP* system that is capable of yielding highly efficient laser diodes emitting at wavelengths of 1.3 μm and 1.55 μm .⁴ These wavelengths correspond to minimum absorption and dispersion losses in glass optical fibers used for optical communications.

However, along with the above mentioned advantages, III-V semiconductor technology has some main drawbacks at the standpoint of OIC applications. Namely, the fabricated semiconductor waveguide devices have high insertion losses (≥ 3 dB) and the integrated optical circuits are of large sizes (≥ 1 cm). Moreover, the device dimensions are large due to the low index contrast of grown layers. And finally, since the fabrication process requires sophisticated technology, the cost of the production is quite high at present.

Lithium Niobate

Between 1972 and 1975 single crystal lithium niobate films were formed by eight different methods.³ Never before had a single material obtained so much attention from material scientists. Fabrication of optical waveguides on *LiNbO₃* substrates became an attractive idea due to its electrooptic and acousto-optic properties as well as its high transparency in the near infrared region. The realization of waveguides was achieved by in-diffusion of titanium into *LiNbO₃* substrates. Maximum refractive index differences between substrate and diffused waveguide are in the range of 10^{-2} - 10^{-3} .⁷ The optical mode size of single mode 1.55 μm device is around 6 - 8 μm which is very similar to the characteristics of single mode optical fibers. This is the reason for very low coupling losses of *LiNbO₃* guided-wave devices (~ 0.5 dB/facet). The fabricated waveguides are reported with low propagation losses too (≤ 1 dB/cm).⁶

The major disadvantages of $LiNbO_3$ technology are as follows: there are difficulties in integration of lithium niobate with other active optical devices which prevents large scale integration, lithium niobates have large device sizes due to the low achievable index contrast, and finally their cost is even more than that of $GaAs$.⁴

Polymers

Another potential material group for the hybrid technology that arised about twenty years ago are polymers. Polymer films are found to be superior to other organic films because of their easy processing and thermal stability.¹³ They are produced by simply dissolving the polymer powder in a solvent and then spin coating it on any type of substrate before being dried in an oven to get rid of the residual solvent. The chemical and mechanical stabilities of the films may have to be improved by further thermal curing or *UV* exposure. Optical waveguide design requires fabrication of multilayers with cladding and core polymers. For that reason control of the refractive index, thickness of the film and its resulting mode geometry structure is achieved by adjusting the chemical composition and the viscosity of polymers. Furthermore, the control of the index of refraction of the amorphous polymer films may be realized by selective photopolymerization in which the ultraviolet light is irradiated through a photomask on plastic polymer film. The index change due to photopolymerization ranges between 0.001 and 0.04, while the refractive indices of polymer films are roughly between 1.3 and 1.8 (at $\lambda = 1.32 \mu m$).⁷

The primary advantages of the polymer films come from their flexibility in the fabrication of films of optical quality and from their compatibility with almost all substrate materials such as *Si*, *GaAs*, *InP*, and *glass*.¹⁴ But this technology has still some serious problems. Decrease of attenuation at $1.55 \mu m$ is difficult to achieve as the minimum propagation loss obtained is $0.5 dB/cm$ for RIE defined strip waveguides.¹³ Although easy to fabricate, the polymer waveguides are very poor in terms of thermal stability compared with that of dielectric waveguides. And finally, the maximum available index change is around 5 % which limits the

extend to which device dimensions can be decreased.

Silicon Based Technologies

Silicon technology was and remains as the most dominant force in electronic integrated circuits which seems that will continue in near future.¹⁵ The work towards implementation of silicon technology in the integrated optics area started ever since the notion of integrated optics was introduced. There are several key factors for this application to be successful. First of all, any candidate technology must provide waveguide structures with low propagation losses. Moreover, it should be compatible with microelectronics device processing and provide high reproducibility with reasonably low cost. The coupling between an OIC and other elements such as optical fibers, light sources, and photodetectors is another important issue of consideration. Si-based technologies offer crucial advantages on all these points, which makes it more attractive over all other competing technologies.

Because waveguides are the most fundamental components used in integrated optical circuits we will continue with focusing on their properties. Historically, the first trend towards constructing optical devices and circuits was to apply dielectric thin-film technology. In application of this technology, two main types of structures have emerged. First one is silica (SiO_2) waveguide on silicon.^{9,16} There are several methods of fabrication of this structure, but we will briefly analyze only one of them. The *silica waveguides on silicon* are fabricated by first depositing a thick glass layer on a high silicon-content glass by flame hydrolysis and then forming the waveguides by standard photolithographic pattern definition followed by reactive ion etching. This technology utilizes index variation obtained by doping SiO_2 with *Ti*, *Ge*, and *P*. The final layer structure has the form of Si (substrate)/ SiO_2 (buffer)/ SiO_2 - TiO_2 (core)/ SiO_2 (cladding). The index difference between the core and cladding layers varies between 0.004 and 0.01. The primary advantages of this technology are the low fiber coupling loss ($\sim 0.05 \text{ dB/facet}$) due to its large core size of $8 \mu\text{m} \times 8 \mu\text{m}$ and the low propagation losses ($\leq 0.1 \text{ dB/cm}$). However the large sizes of the structure raises

problems in LSI and the low index contrast limits the minimum bending radius to $\sim 25 \text{ mm}$.

The other extensively studied structure is $\text{Si} / \text{SiO}_2 / \text{Si}_3\text{N}_4 / \text{SiO}_2$ where *silicon nitride* is used as core layer.¹⁵ The first buffer layer is usually grown by high pressure thermal oxidation (HIPOX) because of the faster oxidation rate needed for $\sim 10 \text{ }\mu\text{m}$ thick oxide layer (the buffer layer must be thick enough to prevent absorption of the guided light by the Si substrate). The core layer is grown by low pressure chemical vapor deposition (LPCVD) of around $0.4 \text{ }\mu\text{m}$ thickness for single mode waveguide with an index of refraction of 1.997 at $\lambda = 0.8 \text{ }\mu\text{m}$. The resulting propagation losses are reasonable ($\leq 0.1 \text{ dB/cm}$ at $\lambda = 0.6328 \text{ }\mu\text{m}$).¹⁷ The main drawback of this technology comes from the small width of the guided mode profile which is typically 100 to 180 nm that prevents efficient interconnection with optical fibers.

Silicon-on-insulator (SOI) is another technology which has been at research stage since 1988. Its main difference comes from the fact that silicon itself is used as a waveguide.¹⁸ This technology was initially developed for isolation applications in electrical circuits. SOI's implementation in integrated optics is possible since silicon is transparent in the near infrared region of $\lambda > 1.2 \text{ }\mu\text{m}$. The fabrication of the structure is done by growing an epitaxial *Si* layer with index 3.45 on an oxide cladding of index 1.46. The most common and efficient technique used for that purpose is separation of implanted oxygen (SIMOX) which implants oxygen of high doses ($\sim 10^{18} \text{ cm}^{-2}$). The structure is annealed at high temperature to form buried SiO_2 layer in silicon wafer. In the waveguides fabricated by this method the vertical direction confinement is step like and the lateral patterning makes use of rib configuration. The thickness of the SiO_2 layer is in the range of few hundred nanometers and that of silicon top layer (used as the guiding layer) varies in a wide range between 200 nm up to any thickness. Reported optical losses of SOI waveguides vary between 0.1-1 dB/cm (depending on the geometrical structure) and the insertion loss values are of the order of 1-2 dB/facet, which at this point happen to be the major drawbacks of this technology.¹⁹

Silicon Oxynitride Technology

As described throughout the section, in the development of integrated optics components, several approaches for different materials and technologies have been used. There are some strong requirements for the devices for optical communication such as low propagation loss, efficient fiber-to-chip coupling, low insertion loss, small bend radii, and low fabrication cost, that should be met for successful realization of the technology. Most of the materials used in integrated optics so far are classified as either low contrast (e.g. silica, LiNbO_3 , polymers) or high contrast (e.g. InP, SOI).²⁰ The satisfaction of all of the above mentioned requirements simultaneously for both of these systems is rather difficult if not impossible. In the low contrast systems the fiber-to-chip coupling efficiency is excellent due to the large size of the single mode waveguides, but they have low integration density since large bend radii (10-30 mm) are required for low loss operation. On the other hand, high contrast systems allow very small bending radii (down to 0.15 mm) with low losses, but efficient fiber-to-chip coupling is difficult to obtain due to small waveguide dimensions.²¹

In recent years, growing attention has been paid to silicon oxynitride (SiO_xN_y or SiON for short) as a potential material for integrated optics.²² This attention has been motivated mainly by its excellent optical properties such as low absorption losses in the visible and near infrared. SiON combines the dielectric properties of SiO_2 together with good chemical inertness and low permeability of Si_3N_4 . In addition, the index of refraction of SiON layers can be easily adjusted continuously over a wide range between 1.45 (SiO_2) - 2.0 (Si_3N_4) which comes to be very attractive property that allows fabrication of waveguides with desired characteristics of fiber match and compactness.^{23,24} Moreover, the growth of SiON layers on silicon substrate is done by well established standard silicon integrated circuit processing technology which is also a key point towards low cost mass production.

Amorphous silicon oxynitride layers are deposited by various techniques. The most used growth technologies are plasma enhanced chemical vapor deposition (PECVD) and low pressure chemical vapor deposition (LPCVD). The PECVD

process is found to be more efficient in controlled deposition of films with refractive indices below 1.7.²⁰ The deposited layers have good uniformity of the refractive index and layer thickness with run-to-run reproducibility.

The waveguides fabricated by this technology have the following structure: silicon is used as a substrate and SiO₂ buffer layer is thermally grown on it to act as a buffer layer for the SiON core film on top of which SiO₂ is deposited to provide symmetric structure.²⁵ The reported propagation losses of the waveguides vary between 1.5-10 dB/cm ($\lambda = 1.55 \mu\text{m}$) for as deposited SiON layers and are reduced to 0.2-0.36 dB/cm for thermally treated films.^{20,21,26-28} Finally, waveguide bends with radius of curvature of 1.5 mm are also reported..²⁹

1.3 Aim of This Study

The main goal of this thesis work is to analyze the optical properties of silicon oxynitride layers for use in low-loss optical waveguides, in terms of suitability, process control and material properties. The processing facilities in our research laboratory are employed throughout the study which proves that the used technologies are standard and readily available. In the following chapters the deposition, material characterization, its treatment towards loss minimization, and finally the fabrication and characterization of single-mode waveguides are described.

In Chapter 2 the process of silicon oxynitride growth by PECVD is analyzed. Chapter 3 includes characterization of the material structure and composition with infrared spectroscopy, together with an annealing study of SiON layers performed in order to minimize the optical losses which are caused by vibrational overtones of the N-H bonds incorporated into the film.³⁰ In Chapter 4 the process of waveguide design and fabrication is monitored. The characteristics such as propagation loss and insertion loss are treated in Chapter 5. Finally, Chapter 6 bears the concluding remarks about this work together with the future plans of study on SiON based technology.

Chapter 2

Deposition Process of SiON Films

This chapter is devoted to the process of fabrication of the "key" material, silicon oxynitride, and to its detailed structural and compositional analysis. Initially, the PECVD reactor and basic CVD processes are explained, after which the characterization study on the growth processes of silicon oxide, nitride, and oxynitride is presented. Finally, the structure and composition of the grown materials are analyzed in detail by making use of ellipsometry and infrared spectroscopy techniques.

2.1 Plasma Enhanced Chemical Vapor Deposition

Chemical vapor deposition has been used widely in the area of microelectronics for growth of layers in several applications such as insulation, masking, optical waveguiding, etc.. It can be defined as a process where thin solid films are grown as a result of chemical reactions of the precursor gases. The PECVD technique in particular, has received wide application in last few decades.

2.1.1 Reactor Properties

The major advantage of plasma enhanced (or plasma-assisted) CVD comes from its capability of growing films at relatively low temperatures (150-350 °C).³¹ In conventional CVD processes the standard temperature of operation is 700-900 °C, which limits its applicability in the structures where metalization features or other temperature sensitive components are involved. The operation at such low temperatures for PECVD technique is possible on the behalf of the addition of electrical energy to the environment resulting in glow discharges.

The system used in our experiments is a parallel-plate type *Plasmalab 8510C* reactor (see Figure 2.1). The radio frequency generated glow discharge (plasma)

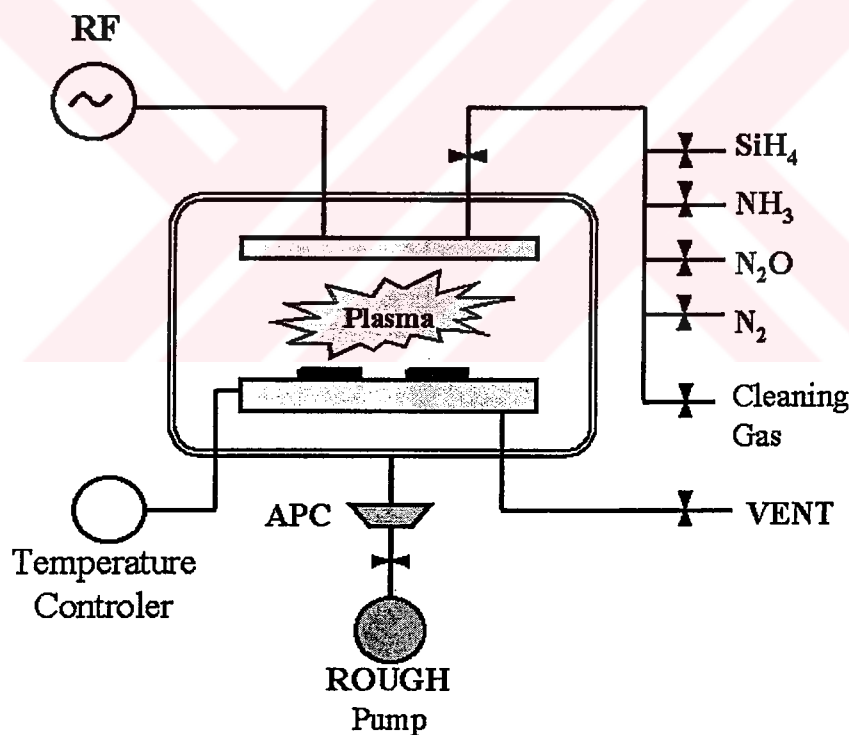
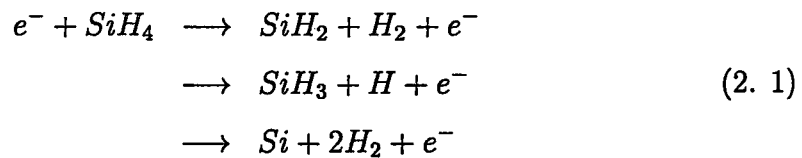


Figure 2. 1: PECVD Reactor Structure

takes place between two electrodes which are separated by 2 cm. The plates are of 24 cm diameter and the RF power establishing the plasma is applied to the upper electrode while the samples are placed on the bottom grounded electrode which

can be heated up to 400 °C. The system can be operated at a pressure range of 0.1-10 Torr. Applied RF frequency is 13.56 MHz (allocated by international authorities as one not to interfere with other communication bands) and the applied RF power can have values up to 300 Watts. The available gases that can be used as precursors for film growth are silane (2% SiH₄/N₂ : diluted in nitrogen because of being highly unstable at room temperature and tendency to burn in case of exposure to air), ammonia (NH₃), and nitrous oxide (N₂O). The structure of the films grown by this technique is amorphous in nature and is to be discussed in a while.

As it was mentioned, the films in our structure are grown by making use of an intermediate state, glow discharge, primary aim of which is to produce chemically active species. Glow discharge consists mainly of ionized gas which contains equal volume concentrations of positively and negatively charged ions and electrons. The electron energy takes values between 1-20 eV and electron density varies from 10⁹ to 10¹²/cm³.³² The first step in the process of deposition of the film is generation of reactant species. Firstly, energy of the applied electric field is coupled to the gas via the kinetic energy of few electrons which ionize and dissociate gas molecules through impact reactions. Thus, new electrons are produced and the process continues in avalanche manner and discharge begins. As an example, below some of the possible electron impact reactions for silane (SiH₄) are given.



The process is observed during the deposition as a glow emitted from the plasma, which is a result of electronic transitions taking place. During this process, electrons are lost to the electrodes and walls due to their high mobility, thus creating a region which is negatively charged compared to the plasma region. This region does not glow and is called "dark region" or sheath and is of thickness of 0.1-10 mm. Electrons are constantly supplied by secondary electron reactions

and impact of positive ions on the electrodes. For stability concern the number of generated and lost electrons should be same.

Another important parameter for the stability of the process is pressure. When it is less than 0.1 torr the mean free path of the electrons and gas molecules increases which results in a decrease of collision's number, thus lowering the rate of dissociation and ionization of gas molecules. On the other extreme, when the pressure is greater than 5 torr, the molecular collision rate increases and plasma again becomes unstable. These two regions of operation should be avoided since they result in nonuniformity and bad control of thickness, index of refraction, and composition of the grown layers. The pressure value used throughout our experiments is 1 torr and is optimized with the other variables resulting in good quality films.

During the process of deposition, after reactive atoms, molecules, and ions are formed, an adsorption of them on the surface site of the substrate or film follows. They have high sticking coefficients and form strong bonds to the surface. After adsorption these radicals are in the form of randomly oriented clusters, which tend to migrate along the surface until forming a continuous film. Finally, the gaseous by-products of the reactions are desorbed from the surface and are pumped out from the chamber. The mentioned property of migration of the clusters allows growth of layers with very good quality. The chemical composition of the films is rather nonuniform, with a possible short range ordering only. Because of this, they are classified as amorphous films and sometimes the process itself is called plasma polymerization.

The process of deposition of solid films on a substrate by using gaseous precursors involves several complicated steps. In a rather simplified approach to CVD process, there are two extreme cases of film deposition conditions. First case is observed at conditions of low chamber pressure and high RF power. The process is called *mass transfer limited reaction*, since the number of reactive species arriving at the surface is less than the reaction rate can handle. The other extreme is called *reaction limited process* and is observed at low RF power and low temperature. For efficient growth characteristics of the desired films,

the optimum deposition conditions have to be between the two extremes and a careful optimization of the reaction chamber conditions have to be conducted.

2.1.2 Basic Characterization Techniques of the Grown Layers

Having analyzed the deposition process conditions for the layers grown throughout this study in basic terms, we can proceed with a short description of the characterization methods used in the analysis of the structures obtained.

There are essentially three parameters of interest that we would like to measure and control for the deposited films. These are thickness of the layer together with its uniformity, index of refraction, and its structural composition. For the measurement of the thickness of the film we have two alternative methods available in our research laboratory. First is a stylus profilometer *Sloan Dektak 3030ST*. By this method, film of any thickness can be measured, provided that necessary step height is produced. In our experiments this type of measurements were performed at regular intervals for verification purposes. The step height was produced by masking part of the film with a photoresist layer and then chemically etching the other part with *1:10 HF* solution.

The other alternative method is to use an ellipsometer. An automated *Rudolph Research/AutoEl III* ellipsometer has been employed in this study. The method is efficient in that it is rapid and nondestructive. Here a monochromatic beam of elliptically polarized light of 632.8 nm wavelength is reflected from the surface of the film as well as from the substrate-film interface (see Figure 2.2). These two components experience different amount of phase shift after reflection and also have different amount of reflection coefficients. Then, analysis of changes in intensity, phase, and polarization of the reflected light gives the desired values of film thickness and refractive index. The process and theory of this analysis is beyond the scope and interest of this study and thus will not be treated. The values for accuracy of measured thickness and refractive index of the film depends on the film-substrate system and on the film thickness. Nevertheless, the accuracy

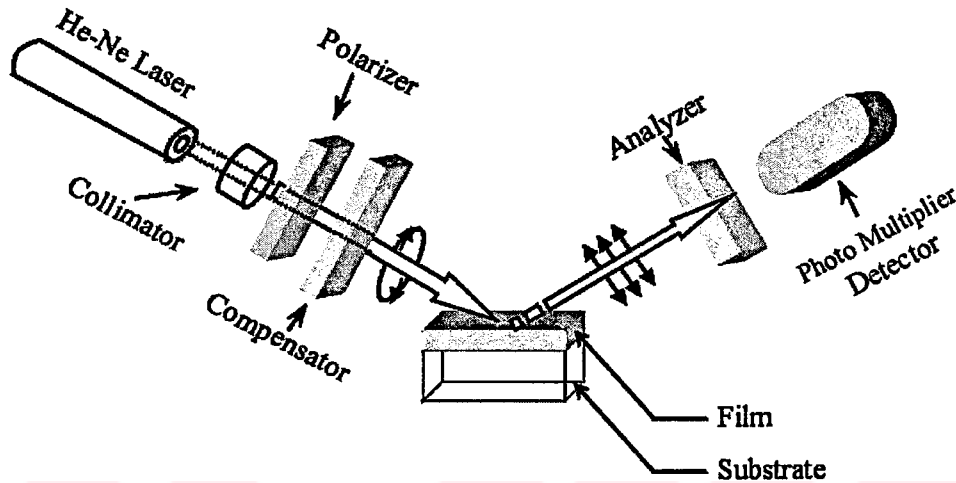


Figure 2. 2: Schematics of a Typical Research Ellipsometer

values typical for silicon oxide films are $\pm 10 \text{ \AA}$ for thickness and ± 0.01 for index of refraction.

The final property of interest is the chemical composition of the grown layers. Its investigation is performed using infrared spectroscopy, in particular Fourier Transform Infrared (FTIR) spectroscopy in the mid-infrared region which will be described in detail in section 2.5.

2.1.3 Preparation of the Samples for Processing

As a substrate material for the grown films we use 3" diameter (100) silicon wafers. Due to cost considerations and the small-size requirements of the processing tools, cleavage of the wafer into smaller samples is performed. The average size of the used samples was $\sim 1.5 - \sim 2 \text{ cm}$ and they were rectangularly shaped due to crystal orientation. The cleavage of the wafers can be done in two ways, depending on the desired quality of the facets. If the optical quality is not a concern, the cleavage

is usually done with a diamond-tipped tool by scribing the wafer. Otherwise, a cleavage through a crystal plane should be induced by creating a small scribe and propagating it along the crystal. This type of cleaving is especially needed after the growth of a layer which is aimed to guide light. The planar cleavage of the substrate results in the same quality facets of the film too.

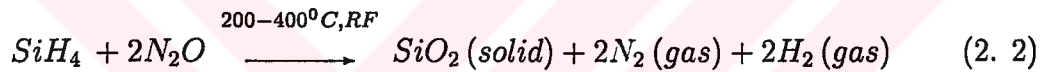
Once the substrate samples are prepared, care must be given to their cleanness. In our case, it is extremely important that the surface of the silicon substrate be clean, since the quality of the film to be grown on it will be affected by any contamination. The first precautionary measure is to perform all processing steps in a clean environment. Thereby, our fabrication processes were done in class 100 and class 10 000 clean rooms. Even in clean room ambient, samples and substrate need further cleaning. The reason for this is the contamination resulting from physical handling, airborne bacteria, leftover of lithographic processes, etc.. In our research laboratory the removal of the contaminant molecules and particles from sample surfaces is done via wet-chemical cleaning process. The first step involves immersion of the samples into boiling trichlorethane (TCA) and processing it for 3 minutes at $\sim 70^{\circ}\text{C}$. Next, the samples are kept in acetone (ACE) for 5 minutes at room temperature after which they are put into an isopropanol (ISO) bath at $\sim 70^{\circ}\text{C}$, again for 3 minutes. Finally, the samples are rinsed in deionized (DI) water before having them dried by blowing pressurized nitrogen (N_2) which smoothly removes it.

2.2 PECVD of Silicon Oxide Films

The physical properties of the silicon oxynitride films are expected to be between that of silicon oxide and silicon nitride. Therefore, monitoring of the compositional characteristics of the mentioned layers should result in a better control of the SiON film properties. For this reason, we allocate the first part of our analysis to a study of the silicon oxide films.

2.2.1 Deposition Parameters

The plasma enhanced deposition of silicon oxide results from the reactions taking place between 2% SiH₄/N₂ and N₂O which in an overall have the following form³⁴



However, the stoichiometry of the grown layers deviates from SiO₂ being SiO_x due to certain amount of hydrogen and nitrogen incorporated into the film, resulting in an index larger than 1.46.

In the experiments to be described in the next subsection, the process parameters of Table 2.1 were used for the deposition of SiO_x layers.

Silane (2% SiH ₄ /N ₂) Flow Rate	180 sccm
N ₂ O Flow Rate	25 sccm
Process Pressure	1000 mTorr
RF Power	10 W
Temperature	250 °C

Table 2.1: Process Parameters for SiO_x Growth #1

2.2.2 Effects of Sample Size

Since we have to use samples of relatively small sizes, the first study done was towards the analysis of the effects of the sample size on the uniformity of the refractive index and thickness of the deposited layers. Thus, we could determine the minimum size which will be "safe" in terms of uniformness.

For this analysis, substrates of several sizes were investigated: $s1$ - 55 mm x 20 mm, $s2$ - 30 mm x 31 mm $s3$ - 20mm x 21 mm, $s4$ - 15mm x 15 mm, and $s5$ - 10mm x 11 mm. Silicon oxide layer was deposited on them in a single run using the parameters given in Table 2.1 with a process time of 20 minutes. The thickness and refractive index values were measured with an ellipsometer starting at ~ 5 mm from the edge of a sample and repeating the measurements in 5 mm intervals over a central line through the samples. The results are given in Figures 2.3-2.5 for the samples $s1$, $s3$, and $s5$.

The measurements showed that the average film thickness for samples $s1$ & $s3$ was ~ 6100 Å with a standard deviation of ~ 40 Å over the whole film, while for sample $s5$ the corresponding value was 6276 ± 30 Å. The average refractive index value for all the samples didn't differ from each other and was measured to be 1.54 ± 0.01 . The results obtained from five different sized samples suggest that edge effects become important when dimension along one side is as small as ~ 1 cm. The film growth rate is found to be greater at the edges of the samples and if

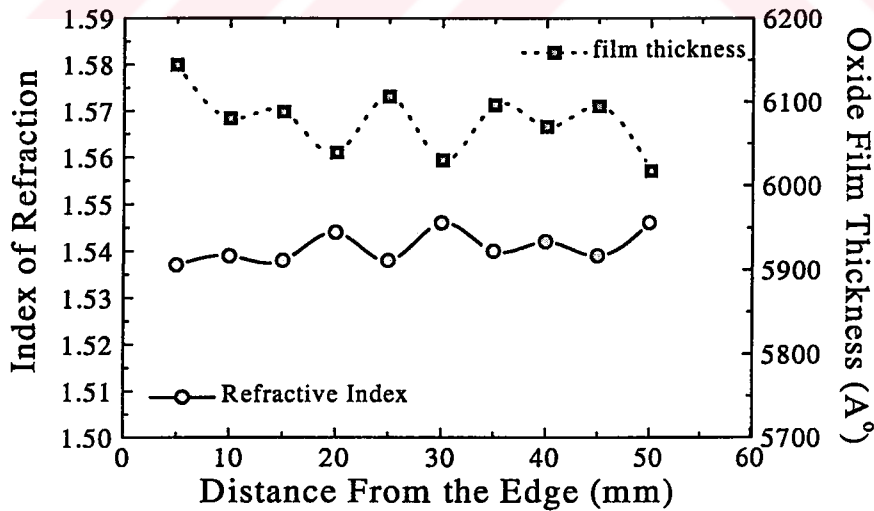
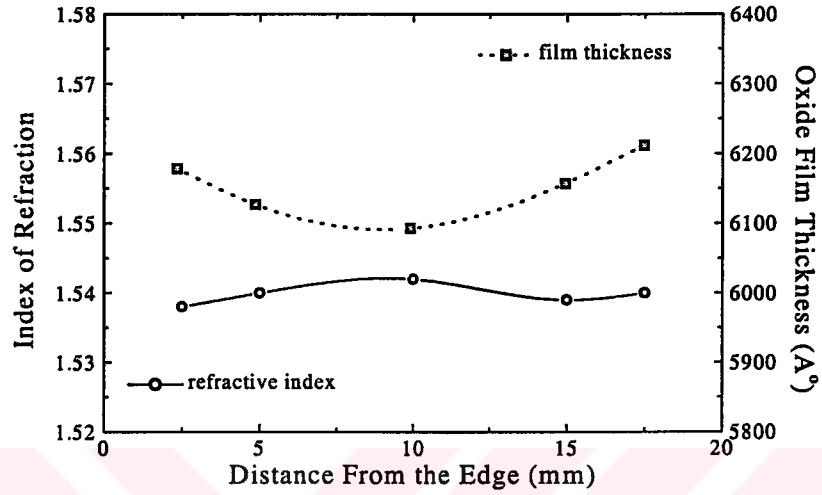
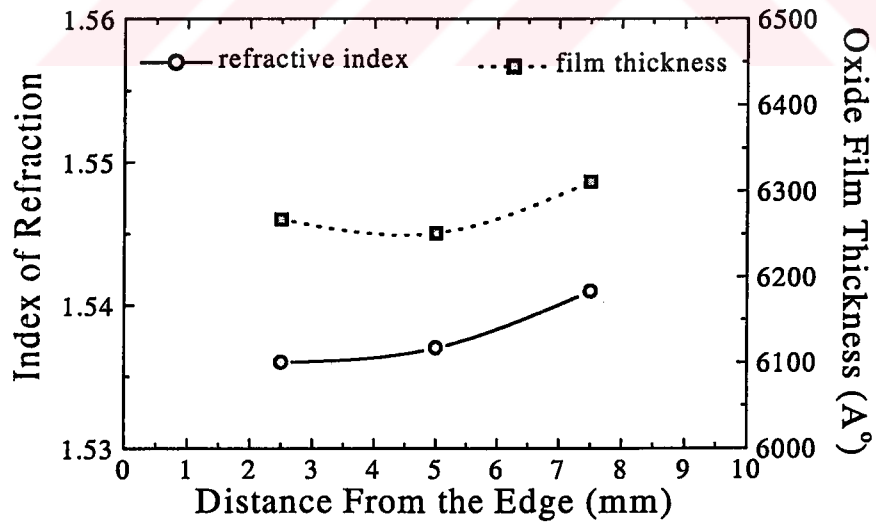


Figure 2. 3: Index and Thickness Variations Over the Sample $s1$ (55mm x 20mm)

Figure 2. 4: Index and Thickness Variations Over the Sample s_3 (20mm x 21mm)Figure 2. 5: Index and Thickness Variations Over the Sample s_3 (10mm x 11mm)

their size is below the critical value, the overall rate appears to be larger than it is in larger samples. In turn, this results in poor control of the layer thickness. This effect was eliminated in our samples by choosing dimensions of ≥ 1.5 cm x 1.5 cm.

Similar studies were done in irregular time intervals and in a nonsystematic way for grown silicon nitride and oxynitride films, yielding identical results and proving the sample size of 1.5 cm x 1.5 cm to be the largest "safe" from the edge effects.

2.2.3 Refractive Index Characterization

After determination of the minimum sample size, our next concern is to grow silicon oxide films and analyze their thickness and refractive index properties. For this purpose, various films were grown at 250 & 350 °C with the process parameters given in Table 2.2. Keeping the flow rate of silane constant, that of

Silane (2% SiH ₄ /N ₂) Flow Rate	180 sccm
N ₂ O Flow Rate	25-300 sccm
Process Pressure	1000 mTorr
RF Power	10 W
Temperature	250 °C, 350 °C

Table 2.2: Process Parameters for SiO_x Growth Series #2

N₂O was changed between 25 and 300 sccm. In this way it was shown that the index of the films can be changed between 1.56 and 1.47 (see Figure 2.6). The value of the index of refraction reaches saturation after a flow rate of N₂O of about 100 sccm. The fitted exponential function for 250 °C deposition has the form of

$$n = 1.464 + 0.071 \times e^{-\frac{N_2O(sccm)-25}{30.340}} \quad (2.3)$$

and that for 350 °C was found to be

$$n = 1.473 + 0.081 \times e^{-\frac{N_2O(sccm)-20}{16.037}} \quad (2.4)$$

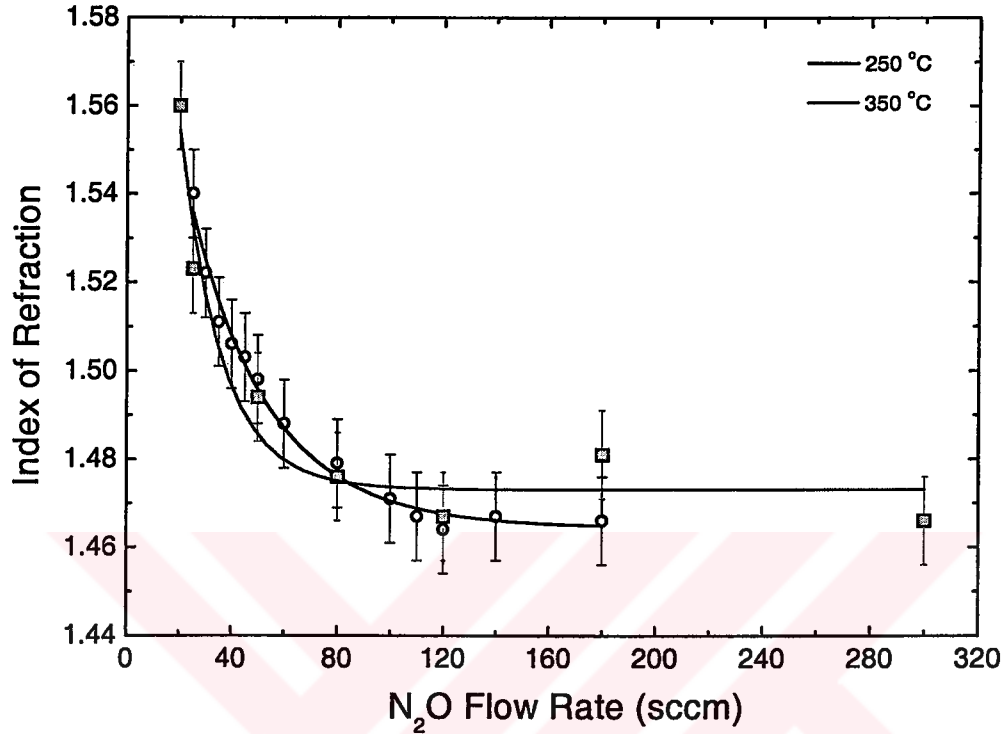


Figure 2. 6: Variation of Index of Refraction of SiO_x Films With Change of Flow Rate of N_2O

In addition, 100 sccm flow value is the point where the deposition rate saturates too, after a steady increase. The observed behavior can be explained by the large amount of nitrogen incorporated into the film and the film's itself being silicon rich in the region where N_2O flow is less than 100 sccm. After this point, silicon oxide formation begins to dominate over silicon nitride formation since it is now available in excess amount and has greater affinity for reacting with silane.

The above characterizations were performed in 250 °C and 350 °C in order to compare the film qualities. It has been found that the growth rate decreases as the process temperature is increased. For example, for N_2O flow of 25 sccm the growth rate at 250 °C is 340 Å/min and for 350 °C it is 290 Å/min. This is a direct indication that the films grown at higher temperatures are denser and contain less microvoids, which was also verified by etch rates of these layers. Another note is about the relation of the N_2O flow to the film density. As it is

increased, the reaction rate (thus the growth rate) increases which results again in a lower density of the layers.

After analysis of the index and thickness characteristics of SiO_x layers, next step would be to determine their compositional properties, the study of which is allocated for the next chapter.

2.3 Deposition of Silicon Nitride Films

Silicon Nitride, other than oxide, is a material, properties of which would be of interest as we expect it to be the other extreme for silicon oxynitride layers to be grown. The stoichiometric ratio of silicon nitride is known to be 3/4 (Si_3N_4), however again PECVD silicon nitride films deviate from this because of the incorporation of hydrogen into the layers. The overall reaction describing the process can be written as³⁴



The process conditions used for nitride deposition in our experiments are given in Table 2.3. Growth processes were carried out at 350 °C due to the advantages

Silane (2% SiH_4/N_2) Flow Rate	180 sccm
NH_3 Flow Rate	5-45 sccm
Process Pressure	1000 mTorr
RF Power	10 W
Temperature	350 °C

Table 2.3: Process Parameters for Si_xN_y Growth

such as denser film and higher index of refraction, explained for the case of silicon oxide films. Moreover, at this temperature incorporation of hydrogen into the layer is reported to be less which is also advantage fitting our requirements.³¹

The characterization of the index variation of the grown nitride films was done by changing the flow rate of NH_3 and keeping that of silane constant at a value of 180 sccm (see Figure 2.7). The layers were deposited in 30-minute processes

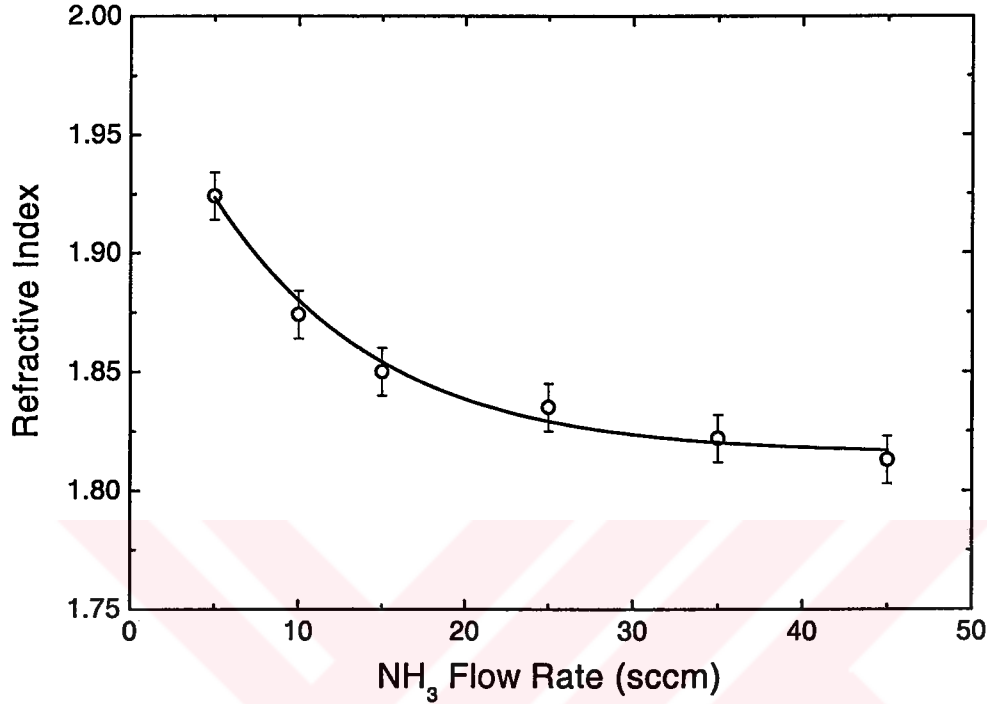


Figure 2. 7: Variation of Index of Refraction of Si_xN_y Films With Change of Flow Rate of NH_3

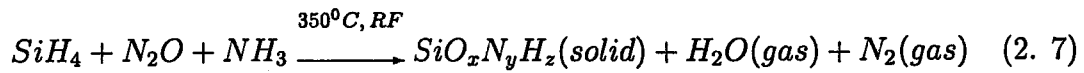
with resulting film thicknesses of $\sim 2800 - 3100 \text{ \AA}$. A regular trend of decrease of the growth rate with increasing NH_3 flow between $120 \text{ \AA/min}$ and $95 \text{ \AA/min}$ was observed. The resulting index values ranged between 1.93 and 1.82 and the fitted function had the following form:

$$n = 1.815 + 0.108 \times e^{-\frac{\text{NH}_3(\text{sccm})-5}{9.745}} \quad (2. 6)$$

The index of refraction is higher at low NH_3 concentration since the layers are silicon rich and decreases with increasing NH_3 flow due to the increase of the amount of nitrogen and hydrogen in the film composition.

2.4 Growth of SiON Layers

Having investigated the growth characteristics of silicon oxide and silicon nitride films, we have become equipped with the necessary experimental results required for the deposition of silicon oxynitride layers. SiON films were obtained by using silane, ammonia, and nitrous oxide as reactant gases. The net chemical reaction pathway for this process²⁴ is



In our experiments we again held the flow rate of silane fixed at 180 sccm and varied the flow rates of N₂O and NH₃. The grown layers were produced at 350 °C as was done for silicon oxide and nitride (further specifications of the process parameters are given in Table 2.4).

Silane (2% SiH ₄ /N ₂) Flow Rate	180 sccm
NH ₃ Flow Rate	0, 15, 30 sccm
N ₂ O Flow Rate	20-450 sccm
Process Pressure	1000 mTorr
RF Power	10 W
Temperature	350 °C
Deposition Time	15 min.

Table 2.4: Silicon Oxynitride Deposition Parameters for Refractive Index Characterization

Index of refraction and thickness of the samples were measured with an ellipsometer at $\lambda=632.8$ nm. Characterization was done by varying the N₂O flow and holding that of NH₃ fixed at three different values. Overall, the refractive index of the deposited layers could be varied between 1.67 and 1.47. The highest refractive index could be extended to ~ 2.0 , but for our application purposes 1.67-1.47 range was sufficient. A general trend of decrease in the film refractive index with increasing oxygen was observed in all cases (see Figure 2.8), which comes about because of oxygen's greater chemical reactivity compared to nitrogen. In addition, as the flow rate of ammonia was increased, the film index increased

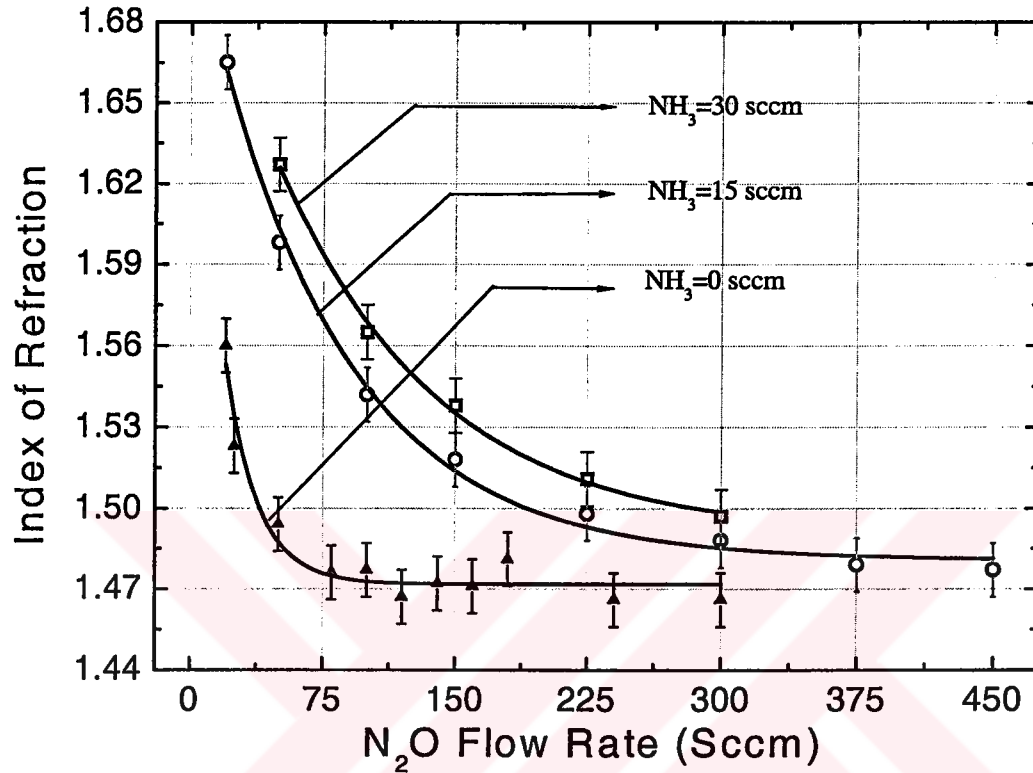


Figure 2. 8: Refractive Index Characterization of Silicon Oxynitride Films as a Function of N₂O and NH₃ Flow Rates

similarly due to their higher nitrogen content, thus gaining more resemblance with the silicon nitride layers.

The film thickness values of the samples varied roughly between 4300 Å and 3000 Å. It was found that increase in nitrous oxide flow rate results in an increase of film growth rate too. Moreover, the deposition rate was observed to be decreasing with an increase of the ammonia flow rate (see Figure 2.9). These properties are attributed again to the oxygen's greater affinity for reacting with silane. Namely, in the first case, as the reactive oxygen concentration increases it begins to dominate in the chemical reactions over nitrogen. As for the decrease of the growth rate with increase in nitrogen concentration in the film, the probability of the nitrogen related bondings now has increased so that

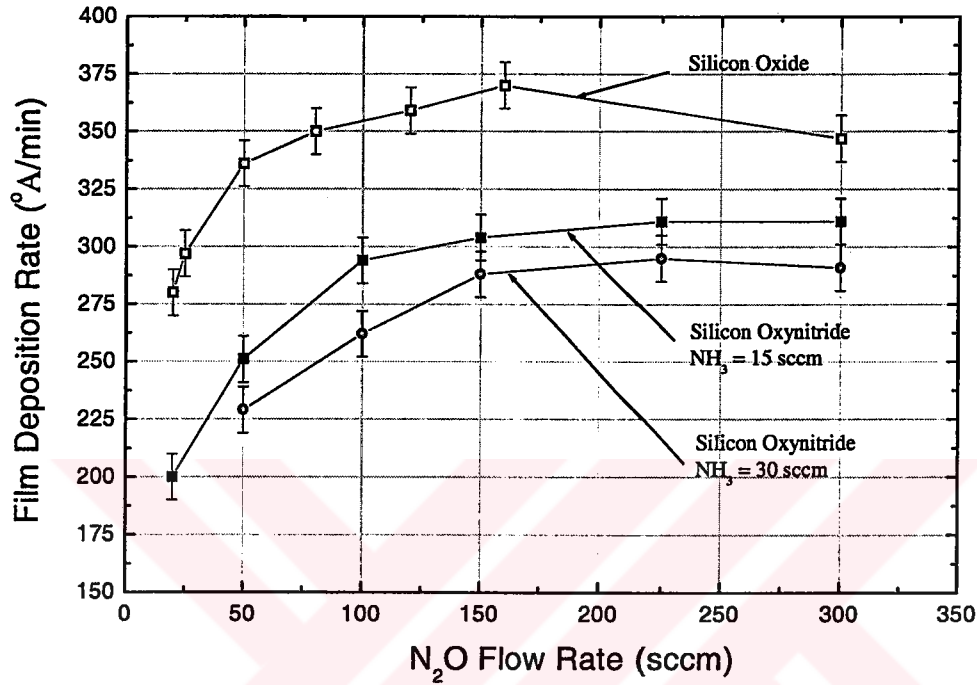


Figure 2. 9: Variation of Film Deposition Rate for Deposited Films a Function of N₂O and NH₃ Flow Rates

nitrogen's concentration in the film increases. Thus, the layer becomes more similar to silicon nitride structured film the growth rate of which is smaller than that of silicon oxide films. In fact, if we consider the growth rates for these films they increase in the following order: silicon nitride, silicon oxynitride, silicon oxide. This is what we would expect, namely a smooth transition of the physical properties of silicon oxynitride from those of silicon oxide to silicon nitride.

Chapter 3

FTIR Characterization

Considering the goals of our work, in addition to characterizing the optical properties of silicon oxynitride films, we would benefit much from learning the internal structure and composition of the grown layers. Thereby, an attempt is made towards relating the material composition to film characteristics such as index of refraction and optical loss. For this aim, a widely-used infrared spectroscopic technique, Fourier Transform Infrared (FTIR) spectroscopy, will be employed. Below, the theoretical grounds of the subject are given, after which the experimental setup is described, and finally the obtained results for the grown layers are presented.

3.1 Theory of Infrared Absorption Spectroscopy

Infrared absorption makes use of the vibration of atoms in molecules. The spectrum is obtained by having radiation pass through a sample and analyzing the absorption features at particular energies, which in turn correspond to the frequency of vibration of a part of a molecule in the structure.

Classical Theory of Molecular Vibrations

In the name of completeness, classical theory of vibration for diatomic molecule with masses m_1 and m_2 connected by a massless spring is briefly reviewed.³⁵ This is the familiar problem of classical harmonic oscillator, where masses move along "molecular" axis and the displacement of each mass from its equilibrium position is represented by x_1 and x_2 (see Figure 3.1). The bond is assumed to

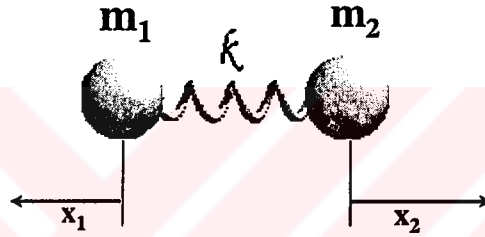


Figure 3. 1: Diatomic Molecule Oscillator : Classical Picture

obey Hooke's law, thus it is possible to write the equations of motion for the two masses as

$$F(x_2 - x_1) = m_1 \frac{d^2 x_1}{dt^2} \quad \text{and} \quad -F(x_2 - x_1) = m_2 \frac{d^2 x_2}{dt^2} \quad (3. 1)$$

here the force on the second mass is in minus x direction which accounts for its negative sign. For simultaneous solution of both of the equations we assume a cosine function with a period of $\frac{1}{\nu}$ (in cycles per second) and therefore we have

$$x = \cos\left[\omega\left(t_0 + \frac{1}{\nu}\right)\right] = \cos(\omega t_0 + 2\pi) \quad (3. 2)$$

where ω is a constant. For most general case an amplitude factor A and a phase factor α , to define the angle at $t=0$, is added. Now the trial solution has the form of

$$x_1 = A_1 \cos(2\pi\nu t + \alpha) \quad \text{and} \quad x_2 = A_2 \cos(2\pi\nu t + \alpha) \quad (3. 3)$$

After substitution of these equations into Eqn. 3.1 we obtain conditions for the amplitude ratios in the form of

$$\frac{A_1}{A_2} = \frac{F}{F - m_1 4\pi^2 \nu^2} \quad \text{and} \quad \frac{A_1}{A_2} = \frac{F - m_2 4\pi^2 \nu^2}{F} \quad (3.4)$$

eliminating A_1/A_2 and solving for ν we get

$$\nu^4 = \nu^2 \left(\frac{F(m_1 + m_2)}{4\pi^2 m_1 m_2} \right) \quad \text{and} \quad \frac{A_1}{A_2} = -\frac{m_2}{m_1} \quad (3.5)$$

One solution is $\nu = 0$, which corresponds to translation in x direction, and the other is

$$\nu = \frac{1}{2\pi} \sqrt{F \left(\frac{1}{m_1} + \frac{1}{m_2} \right)} = \frac{1}{2\pi} \sqrt{\frac{F}{\mu}} \quad (3.6)$$

where μ is the reduced mass. Substituting kx for the force, the potential energy of the system can be obtained to be

$$U = \frac{kx^2}{2} \quad (3.7)$$

and the vibrational frequency is given by

$$\nu = \frac{1}{2\pi} \sqrt{\frac{k}{\mu}} \quad (3.8)$$

Thus, the force constant can be calculated for given frequency and reduced mass and vice versa.

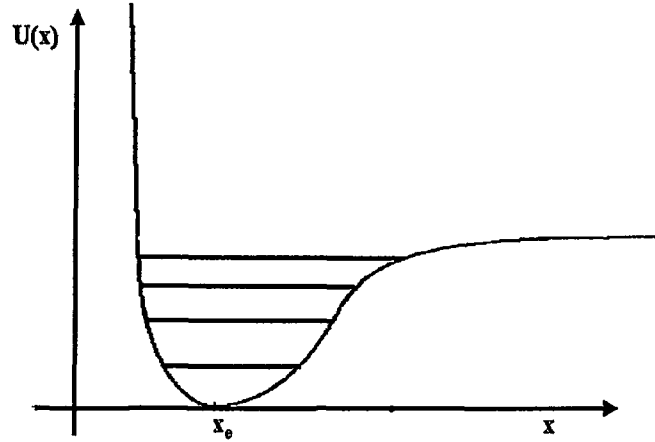
Quantum Mechanical Treatment

A more accurate model for the infrared absorption of molecules is obtained by a quantum mechanical approach. Again a harmonic oscillator is used as a model, where Hamiltonian function is of the following form

$$\hat{H} = -\frac{\hbar^2}{2\mu} \frac{d^2}{dx^2} + 2\pi^2 \nu^2 \mu \hat{x}^2 \quad (3.9)$$

where ω is replaced by $2\pi\nu$.

Once more, concentrating on a simple diatomic molecule, we note that the potential function is not parabolic in shape (see Figure 3.2) but is approximately



Şekil 3. 2: Potential Energy curve for a Diatomic Molecule

so only in the vicinity of the equilibrium internuclear distance x_e . Solving the Schrödinger equation for this exact function proves to be difficult, thus an extent should be determined to which the approximation will be valid or to which it can be improved. For this purpose the exact potential energy function $U(x)$ is expanded in Taylor series about the equilibrium point x_e in the following manner

$$\begin{aligned}
 U(x) \simeq U(x_e) &+ \left(\frac{dU}{dx}\right)_{x_e}(x - x_e) + \frac{1}{2}\left(\frac{d^2U}{dx^2}\right)_{x_e}(x - x_e)^2 \\
 &+ \frac{1}{3!}\left(\frac{d^3U}{dx^3}\right)_{x_e}(x - x_e)^3 + \dots
 \end{aligned}
 \tag{3. 10}$$

The first term in the expansion is the electronic energy at equilibrium point being constant, the next term is zero since $(\frac{dU}{dx})_{x_e}$ is zero at the minimum, x_e , and finally the only contribution that looks to improve the function comes from the third term. If all the other terms are again neglected as giving small contributions only, then $U(x)$ is approximated by a harmonic potential and the resulting Schrödinger equation can be solved in a standard way.

The most important outcome of the quantum mechanical approach is the

discrete energy spectrum given by

$$E_v = (v + \frac{1}{2})h\nu \quad v = 0, 1, 2... \quad (3. 11)$$

where

$$\nu = \frac{1}{2\pi} \sqrt{\frac{k}{\mu}} \quad (3. 12)$$

and μ is the reduced mass of the diatomic molecule. In infrared spectroscopy it is common to express energy in vibrational wavenumbers ($\omega = \frac{1}{\lambda}$) in which case Eqn. 3.11 becomes

$$\frac{E_v}{hc} = G(v) = \omega(v + \frac{1}{2}) \quad (3. 13)$$

This approach gives acceptable results for the lowest vibrational states of diatomic molecules where the energy states are nearly equally spaced and the amplitude of vibrations is small.

Now, we should determine which transitions in a vibrational spectrum are possible for diatomic molecules. This is determined by the interaction of the dipole moment of the molecule with the electric field component of the radiation. Namely, the transition probability is given by the square of the transition dipole moment $\vec{R}_{nm}$, given by

$$\vec{R}_{nm} = \int \Psi_n^* \hat{\mu} \Psi_m d\tau \quad (3. 14)$$

where $\hat{\mu}$ is the electric dipole moment operator and

$$\hat{\mu} = \sum_i q_i \vec{r}_i \quad (3. 15)$$

where the sum is carried over all nuclei and electrons of the molecule, q_i is the charge and $\vec{r}_i$ is their position vector. The transition dipole moment $\vec{R}_{nm}$ can be thought as the oscillating electric dipole moment due to the transition.

For a diatomic molecule of two different species the electric dipole moment μ is non-zero and varies with x . This variation is expressed as a Taylor series expansion about equilibrium point x_e , referring to equilibrium internuclear distance, as

$$\vec{\mu} = \vec{\mu}_e + \left(\frac{d\mu}{dx}\right)_{x_e}(x - x_e) + \frac{1}{2!} \left(\frac{d^2\mu}{dx^2}\right)_{x_e}(x - x_e)^2 + ... \quad (3. 16)$$

Now, for a molecule in a given electronic state, the transition dipole moment takes the form of

$$\vec{R}_{v'v''} = \vec{\mu}_e \int \Psi_{v''}^* \Psi_{v'} dx + \left(\frac{d\vec{\mu}}{dx}\right)_{x_e} \int \Psi_{v''}^* (x - x_e) \Psi_{v'} dx + \dots \quad (3. 17)$$

The first term of this equation is zero because the vibrational wavefunctions for different v (states) are orthogonal. The second term is non-zero if

$$\Delta v = \pm 1 \quad (3. 18)$$

which imposes a vibrational selection rule. Another condition for the nonvanishing of this term is that the dipole moment should be a function of the internuclear distance x . This means that homonuclear diatomic molecules, such as N_2 and H_2 , have zero dipole moments for all bond lengths and therefore show no vibrational spectra. For the higher terms of Eqn. 2.32 we would expect that their contribution is small or can be neglected. If they are neglected the harmonic oscillator model would give us only single vibrational absorption at specific frequency, otherwise the higher terms do give rise to *overtone transitions* of $\Delta v = \pm 2, \pm 3, \dots$ (i.e. $v_0 \rightarrow v_2, v_0 \rightarrow v_3$, etc.) with rapidly decreasing intensities.

The transitions of molecules from excited state to another excited state are discarded due to the small fraction of molecules at these states at room temperature, since the number of population of the v^{th} vibrational level is related to the ground state by the Boltzman factor

$$\frac{N_v}{N_0} = e^{-\frac{E_v}{kT}} \quad (3. 19)$$

The discussed model turned to be useful in predicting some phenomena such as lack of vibrational transition for homonuclear molecules and overtone transitions. However, it has a limitation of being valid only near the region of minimum potential energy. The problem can be overcome by introducing a more accurate function of the potential (as was done by Morse in 1929) which accounts for the anharmonic behavior.

Polyatomic Molecules

A polyatomic molecule consisting of N atoms has $3N - 6$ ($3N - 5$ if it is linear) *normal modes* of vibration.³⁶ Actually, the total vibrational motion of the molecule is resolved into components which consist of mixture of angle-bending and bond-stretching vibrations called to be normal modes (see Figure 3.3). In particular, Schrödinger equation describing the system is separated into $3N$ coordinates, three of which correspond to translation, two or three to rotation, and the rest to vibrations along the normal modes.

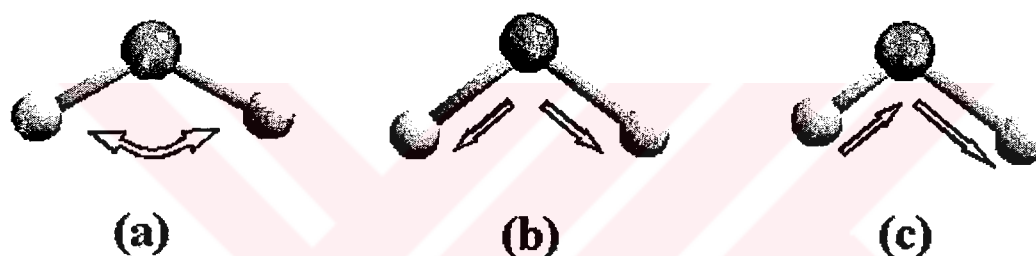


Figure 3. 3: Illustration of (a) bending, (b) symmetric stretching, and (c) antisymmetric stretching vibrations for a polyatomic molecule.

In a normal mode of vibration, all the nuclei move in phase, have the same frequency of vibration, and in general move with different amplitudes. In this motion, the center of mass of the molecule does not move and the molecule as a whole does not rotate. Moreover, any vibrational motion of this polyatomic molecule is expressible in terms of linear combination of its normal modes.

Turning to the quantum mechanical treatment of the molecule, again the vibrations are considered to be harmonic. This approach results in a total energy of the molecule to be

$$G(v) = \sum_i^{3N-6(5)} \omega_i \left(v_i + \frac{1}{2} \right) \quad (3. 20)$$

where ω_i is the wavenumber of the i^{th} mode, v_i is its vibrational quantum number, and the frequency of the normal mode depends on the force constant and reduced mass for the mode.

It should be noted that, again we can have overtone vibrations. In addition, for polyatomic molecules there can be further *combination bands* which come about from two different modes of vibration and add together to show up at higher energy level. That is, in this transition one photon excites two different vibrations simultaneously.

Another note to be mentioned, is that in addition to the basic stretching and bending vibrations, there are terms such as rock, twist, scissors, wag, and breathing that are frequently used in description of the group vibrations of molecules, the illustrations of which are given in Figure 3.4.

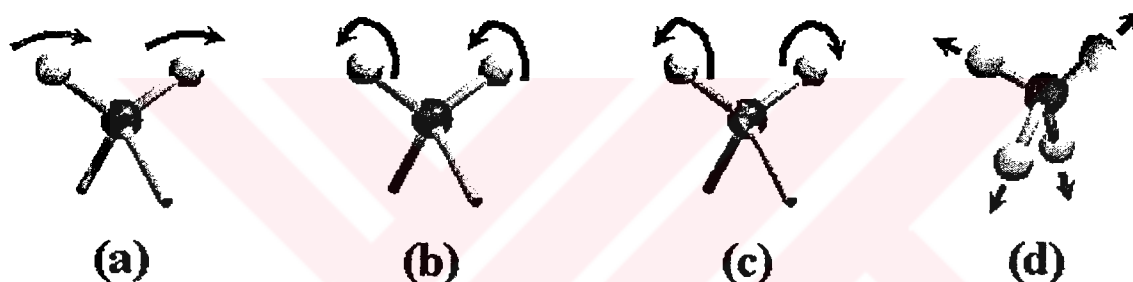


Figure 3. 4: Schematics of some of the additional vibrational terms used for the description of various normal modes : (a) rocking, (b) wagging, (c) twisting, (d) breathing vibrations.

The final remark is about the selection rules for infrared active vibrations. The main rule is that any normal mode will have a vibrational spectrum only if its displacements cause a change in the dipole moment. The process of this determination of the vibrational modes that are infrared active is performed by making use of group theory, i.e. making identification of the symmetry properties of the molecule and the possible vibrations which result in dipole moment change.

3.2 Experimental Setup

After a discussion on the theory of infrared absorption, the next step is the analysis of the experimental tools used in the compositional study of the grown films. Throughout our experiments a Bomem H&B Series FTIR setup has been employed (see Figure 2.14).

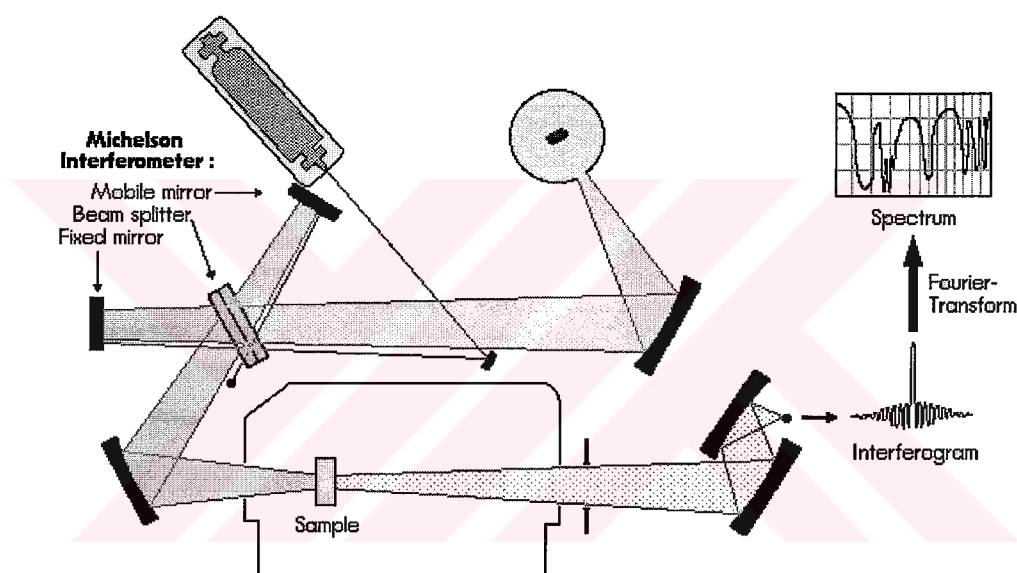


Figure 3. 5: The FTIR Experimental Setup Used for Infrared Transmittance Measurements in the Study

Its basic operation relies on Michelson interferometer, which is built into the spectrometer. The advantages of this technique over the other spectroscopic methods derive from using a large aperture at the signal input and having the entire spectrum at once at the output. Namely, using a large aperture results in a large energy throughput and is called Jacquinot advantage. Moreover, the ability to simultaneously process the entire spectral range in a single scan gives an advantage which is known as Fellgett advantage.

As a source in the spectrometer, a Nerst solid glower is used, which

radiates continuous light in the mid-infrared region in accordance with blackbody radiation. The light then is directed to the beam splitter of the Michelson interferometer, after which it is reflected from two perpendicular plane mirrors one of which is movable. The radiation reaching the detector (pyroelectric deuterium tryglycine sulfate : DTGS) consists of two components that have a path difference of δ and whose magnitude is changed by displacing the movable mirror. The intensity of light measured at the detector, which represents the interference between the two beams, is of the form of

$$I(\delta) = \int_0^\infty B(\omega) \cos(2\pi\omega\delta) d\omega \quad (3. 21)$$

where ω is the wavenumber and $B(\omega)$ is the source intensity at that wavenumber. This expression is also called as *interferogram* and its Fourier transform gives the desired spectrogram of the transmitted intensity as a function of the wavenumber, i.e. $B(\omega)$ given by

$$B(\omega) = 2 \int_0^\infty I(\delta) \cos(2\pi\omega\delta) d\delta \quad (3. 22)$$

The process of this transformation is performed by a computer which also gives the desired spectrum plot.

Having obtained the desired spectrum we shall look at what it does represent. When we pass radiation of intensity I_0 through a sample the transmitted intensity is I . The ratio I/I_0 is called the transmittance of the sample. If we have a sample of thickness t and concentration c we can define *absorbance* which is directly proportional to above quantities as

$$A = \alpha tc \quad (3. 23)$$

where α is a constant of proportionality called as *molar absorptivity* which has dimensions of inverse concentration $\times$ length. The absorbance (A) and transmittance (T) are related by a relation of the form

$$A = \log_{10} \frac{I_0}{I} = \log_{10} \frac{1}{T} \quad (3. 24)$$

which is known as *Beer's law*.

In the obtained IR spectrum the total intensity of a specific absorption band is measured by the integrated intensity, which is carried out over the band. The integrated absorption coefficient is defined by

$$\int_{band} \alpha d\omega \quad (3. 25)$$

and if the band is Gaussian in shape then the relation can be approximated by

$$\int_{band} \alpha d\omega = K\alpha_{max}\Delta\omega_{1/2} \quad (3. 26)$$

where K is a calibration constant specific to the vibration and $\omega_{1/2}$ is the width at half maximum of α .

Summarizing, infrared spectroscopy is a nondestructive technique which provides direct information on the compositional and structural properties of the film, the types of chemical bonds present there, on their impurity content and concentration. In the obtained spectrum of the film the spectral positions of the absorption bands correspond to the vibrational frequency responsible for it, their intensity to the number of such bonds, and their width to the extent of different frequency values related to the different atomic arrangements surrounding the bonds.

3.3 Infrared Analysis of the Grown Silicon Oxide Films

In our FTIR transmittance measurements, the first concern was whether there is a change in the composition of the samples when exposed to air. It was determined that, in a time intervals of few days there was no observable incorporation of oxygen or hydrogen into silicon oxide, nitride or oxynitride layer. In spite of this, usually, their IR spectrum was taken within few hours after growth.

The deposited films were of thicknesses of about $\sim 6000 \text{ \AA}$ with the same growth parameters as in Table 2.2. Four samples were used to monitor the constitutional characteristics of the films. Namely, samples *o1*, *o2*, *o3*, and *o5* with the corresponding N_2O flow rates of 25, 50, 120, and 300 sccm, respectively. Before obtaining the infrared transmittance spectrum of the samples, a background scanning was obtained using the same substrate (double side polished Si (100)). Performing this, it was aimed to eliminate the complications in the spectrum arising from the substrate and air. Detailed parameters of FTIR transmittance measurements are given in Table 3.1.

Scanned Range	6000-250 cm^{-1}
Resolution	8 cm^{-1}
Scan Number	1024
DTGS Detector Gain	B
Interferogram Alignment Ratio	53 %

Table 3.1: Experimental Parameters of the FTIR Transmittance Measurements for Silicon Oxide Layers

Configuration of the measurements was such that the radiation incidence angle is perpendicular. The obtained spectra of the above mentioned samples are shown in Figure 3.6 in terms of absorbance (see Eqn. 3.24). In the figure, the observed vibrational types are enumerated and identified to be as given in Table 3.2.

Before examining the changes in the structure of the films with change of N_2O flow rate, we shall discuss the general characteristics that are common to all of them. All the samples show dominant feature of absorption at $\sim 1050 \text{ cm}^{-1}$

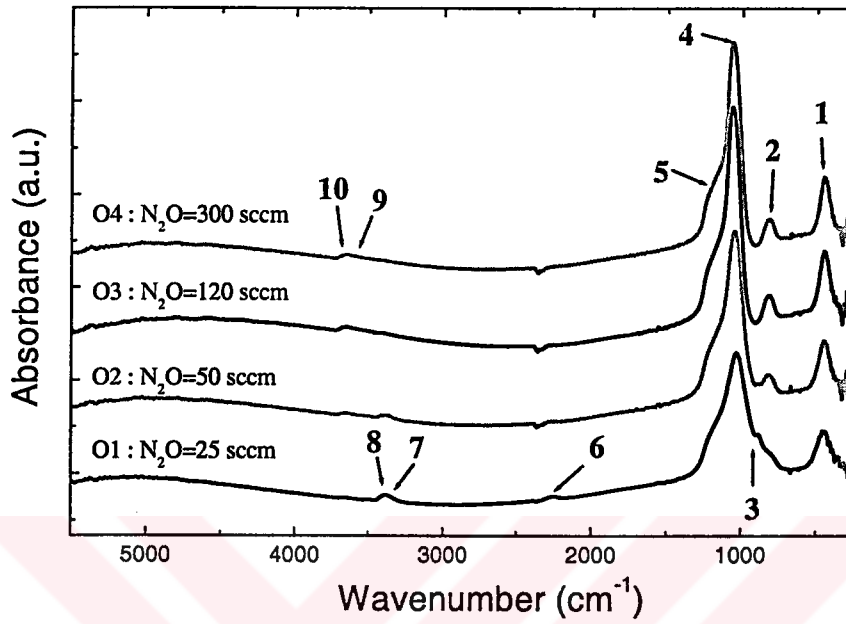


Figure 3. 6: IR absorbance of silicon oxide layers grown with 25, 50, 120, and 300 sccm N_2O flow rate, respectively

Vibration Type	Peak Frequency (cm^{-1})				Reference
	o1	o2	o3	o4	
(1) Si-O rocking	449	448	446	446	37,38
(2) Si-O bending	783*	826	818	819	37,38
(3) Si-H bending	884	...	...	...	38,42
(4) Si-O symmetric stretching	1026	1044	1053	1055	37,38
(5) Si-O asymmetric stretching	1163	1179	1177	1179	37,42
(6) Si-H stretching	2258	2265	...	...	39,40,42
(7) N-H...N stretching	3347	3360	3352	3398	41
(8) N-H stretching	3390	3399	3400	3398	40,41
(9) H-O-H stretching	3493	3499	3528	3542	39,40
(10) SiO-H stretching	3657	3663	3671	3672	39,40
	3586	3595	3623	3628	

Table 3.2: Infrared Vibrations Observed in the Grown Silicon Oxide Samples

which is attributed to the symmetric and asymmetric stretching vibrations of Si-O groups. Si-O rocking vibration is also common to all the samples and is observed at $\sim 450\text{ cm}^{-1}$. While the Si-O rocking vibrational frequency is constant, the Si-O symmetric stretching vibration shifts steadily to higher frequencies from 1026 cm^{-1} to 1055 cm^{-1} as the flow rate of N_2O increases. This is attributed to the fact that, as the oxygen concentration in the film increases it gets more resemblance with stoichiometric SiO_2 , whose corresponding band is at 1080 cm^{-1} .

Another feature that should be mentioned is the Si-H symmetric stretching vibration variations for the samples of consideration. This vibration was detected at 2258 and 2265 cm^{-1} for samples o1 & o2 only, while the Si-H bending vibration was observable only for sample o1. Moreover, for the sample o1, the Si-O bending vibration is observed at quite lower frequency (marked with * in Table 3.2) compared to the others. We attribute this to the coupled vibration of the local structure of Si-O-Si-H, which was also observed by Wolfe *et al.*³⁸ All these samples share the property of being silicon rich, on the other hand, as the oxygen concentration in the film increases, the hydrogen present in the structure tends to form bonds with oxygen itself, instead of silicon or nitrogen. Moreover, the

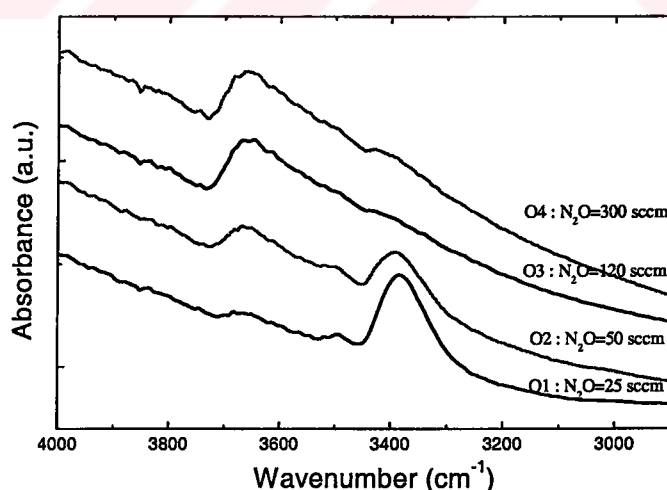


Figure 3. 7: Variation of the O-H and N-H stretching bands with the N_2O flow rate increase.

N-H bond is at the limit of detection for sample o4 and dominates over Si-H in sample o1, which further supports the previous argument. Figure 2.16 shows this in more closer view. The vibration band of N-H bond stretching is of special interest for us, since it is the main cause of the optical absorption at $1.55 \mu m$ wavelength for the optical waveguides that we aim to fabricate. Thereby, we pay special attention to its properties and its evolution for the silicon oxide samples, too. The N-H bond concentration was calculated for the four grown layers by using the technique of Lanford and Rand,⁴³ using the expression

$$[N - H] = \frac{1}{2.303\sigma_{N-H}} \times \int_{band} \alpha(\omega) d\omega \quad (3.27)$$

where σ_{N-H} is the absorption cross section for the N-H bonds, $\int \alpha(\omega) d\omega$ is the normalized absorption area of the band, and $\alpha = \frac{2.303}{t} A$ is the absorption coefficient A being the absorbance and t film thickness. The integration is carried over the band of consideration, which was decomposed using nonlinear curve fitting and assuming that the peaks are in the form of symmetric Gaussians (see Figure 3.8). The results of this analysis are given in Table 3.3.

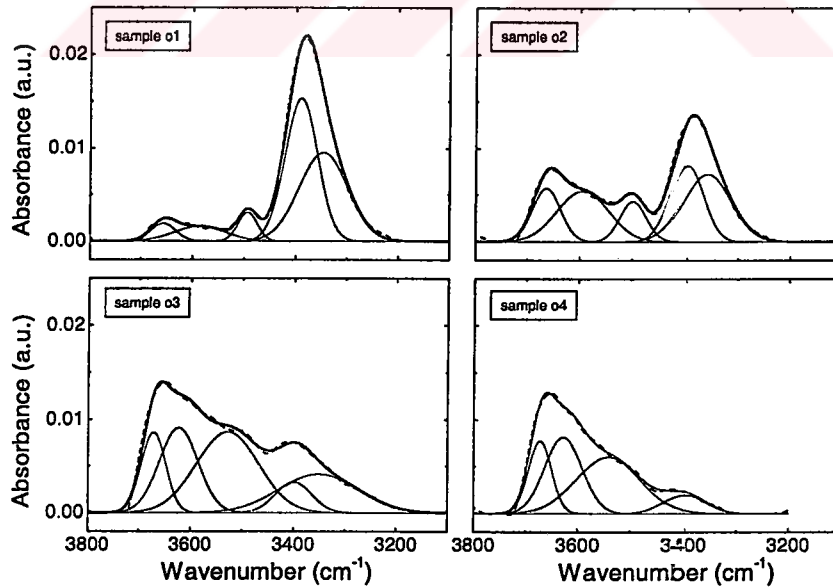


Figure 3. 8: Gaussian deconvolution of the N-H and O-H stretching bands for the four grown silicon oxide samples.

Sample #	Film Thickness ($\text{\AA}$)	Refractive Index	Vibration Type	Peak Wavenumber $\omega(\text{cm}^{-1})$	FWHM (cm^{-1})	Maximum Abs. Coef. $\alpha_{max}(\text{cm}^{-1})$	Normalized Abs. Band Area ($\times 10^5 \text{cm}^{-2}$)	Sum of the Band Area (10^5cm^{-2})	$[N-H]$ (10^{22}cm^{-3})	$[O-H]$ (10^{22}cm^{-3})
O1	5975	1.53	N-H...N	3347	111	367	0.43	0.90	0.74	-
			N-H	3390	74	589	0.47			
			H-O-H	3494	44	119	0.05			
			SiO-H (1)	3586	114	61	0.07			
			SiO-H (2)	3657	56	73	0.04			
O2	6640	1.49	N-H...N	3360	106	251	0.28	0.50	0.41	-
			N-H	3399	71	283	0.22			
			H-O-H	3499	60	150	0.09			
			SiO-H (1)	3595	115	188	0.23			
			SiO-H (2)	3664	64	199	0.13			
O3	7334	1.47	N-H...N	3352	180	129	0.25	0.34	0.28	-
			N-H	3400	82	103	0.09			
			H-O-H	3528	141	271	0.41			
			SiO-H (1)	3623	88	286	0.27			
			SiO-H (2)	3627	56	270	0.16			
O4	6828	1.46	N-H...N	3398	90	66	0.06	0.06	0.04	-
			H-O-H	3542	141	204	0.31			
			SiO-H (1)	3628	83	276	0.24			
			SiO-H (2)	3672	53	262	0.15			

Table 3.3: N-H and O-H Bond Concentration Calculations for Silicon Oxide Films by Using FTIR Transmittance Spectroscopy.

The absorption cross section value $\sigma_{N-H} = 7.4 \times 10^{-18} \text{cm}^2$ used in our calculations was obtained by Lanford & Rand⁴³ through a resonant nuclear reaction and the uncertainty of the calibration technique that they had proposed is reported to be about $\pm 15\%$.⁴⁴ The corresponding calibration factor for O-H bonds however, is not so well defined. This factor was obtained by *Rostaing et al.*⁴⁵ through a fit of the data of elastic recoil detection analysis, accuracy of which is reported to be $\pm 50\%$. In spite of this, we believe that the obtained results can be used safely in the comparison of the four samples, since the change in the O-H concentration in these layers is more than 50%. Nevertheless, care must be given when comparing concentrations of N-H and O-H bonds if absolute values are to be considered.

For the other quantities such as peak wavenumber ω , full width at half maximum $FWHM$, maximum absorption coefficient α_{max} , and normalized absorption band area $\int \alpha d\omega$, they have typical uncertainty values of $\pm 5 \text{cm}^{-1}$, $\pm 5 \text{cm}^{-1}$, $\pm 4\%$, and $\pm 4\%$, respectively.

Looking closely at the results of Table 3.3 we see that the N-H bond concentration of the samples decrease drastically from $0.74 \times 10^{22} \text{cm}^{-3}$ to $0.04 \times 10^{22} \text{cm}^{-3}$ as the oxygen incorporation into the film is increased. The hydrogen atoms now tend to form bonds with oxygen instead, increasing the O-H bond concentration from $0.42 \times 10^{22} \text{cm}^{-3}$ to $1.83 \times 10^{22} \text{cm}^{-3}$. Moreover, the absorption due to N-H...N vibrations arising from deformation of the local structure by forming hydrogen bonds, begins to dominate over the N-H stretching vibration. This can be understood since now the available N-H bonds are surrounded by an increased number of O-H bonds, which in turn cause N-H structure to form hydrogen bonding in increased quantity.

Final evaluation of the grown silicon oxide layers can be made by making a relative comparison of them with the stoichiometric SiO_2 . For this purpose we use a method proposed by *Tsu et al.*⁴² and used by many other authors.^{41,47} In this method, the position of the Si-O (symmetric stretching) peak is used to determine the degree of oxygen content in the SiO_x films by employing a simple

relation of the form

$$x = 0.020(\omega_{Si-O}) - 19.3 \quad (3. 28)$$

according to this relation the oxygen content of our samples is given as

$$\begin{aligned} \text{sample o1} &: SiO_{1.22} \\ \text{sample o2} &: SiO_{1.58} \\ \text{sample o3} &: SiO_{1.76} \\ \text{sample o4} &: SiO_{1.80} \end{aligned} \quad (3. 29)$$

which indicate that our grown SiO_x layers are nearly stoichiometric, with a small amount of hydrogen and nitrogen incorporated into the structure.

Finally, the conclusion that we draw from the compositional study of SiO_x films is that, in the growth of the silicon oxynitride layers using higher flow rates of N_2O should result in lower N-H bond concentrations.

3.4 FTIR Spectroscopy of Silicon Nitride Films

The silicon nitride samples, used for infrared characterization, were deposited by the process parameters given previously in Table 2.3. Four samples *sin1-sin4* were used to trace their structural properties with NH_3 flow rates of 15, 25, 35, and 45 *sccm*, respectively. The samples' film thicknesses were about 3000 Å and their index of refraction varied between 1.85 and 1.81. Again, a background spectrum was obtained just before the IR transmittance measurements, by using identical substrate material as for the deposited layers. This was done in order to increase the effectiveness of its subtraction from the overall spectrum, thus increasing the signal to noise ratio. Other experimental parameters of the FTIR transmittance measurements are listed in Table 3.4.

Scanned Range	6000-250 cm^{-1}
Resolution	8 cm^{-1}
Scan Number	512
DTGS Detector Gain	B
Interferogram Alignment	51 %
Substrate Material	Si double side polished (100)

Table 3.4: Experimental Parameters of the FTIR Transmittance Measurements for Silicon Nitride Layers.

The absorbance spectrum of the above samples is given in Figure 3.9, where the characteristic vibrations are enumerated and identified to be as given in Table 3.5.

The obtained spectrum constitutes mainly of three bands. The first one with strongest features is composed of Si-N breathing ($\sim 470 \text{ cm}^{-1}$), Si-H rocking ($\sim 670 \text{ cm}^{-1}$), Si-N stretching 1 & 2 ($\sim 850 \text{ cm}^{-1}$ & 980 cm^{-1}), and N-H bending ($\sim 1180 \text{ cm}^{-1}$) vibrations. An interesting trend in this band is the shift of the Si-N stretching vibration frequencies to higher values. The second band observed is at ($\sim 2200 \text{ cm}^{-1}$) and is due to Si-H stretching vibrations. This band is resolved into two different components Si-H(N_2Si) and Si-H(N_3), accounted for by the variation in the local structure surrounding the Si-H bonds. The final feature is that of N-H stretching band, resolved into three different components as seen

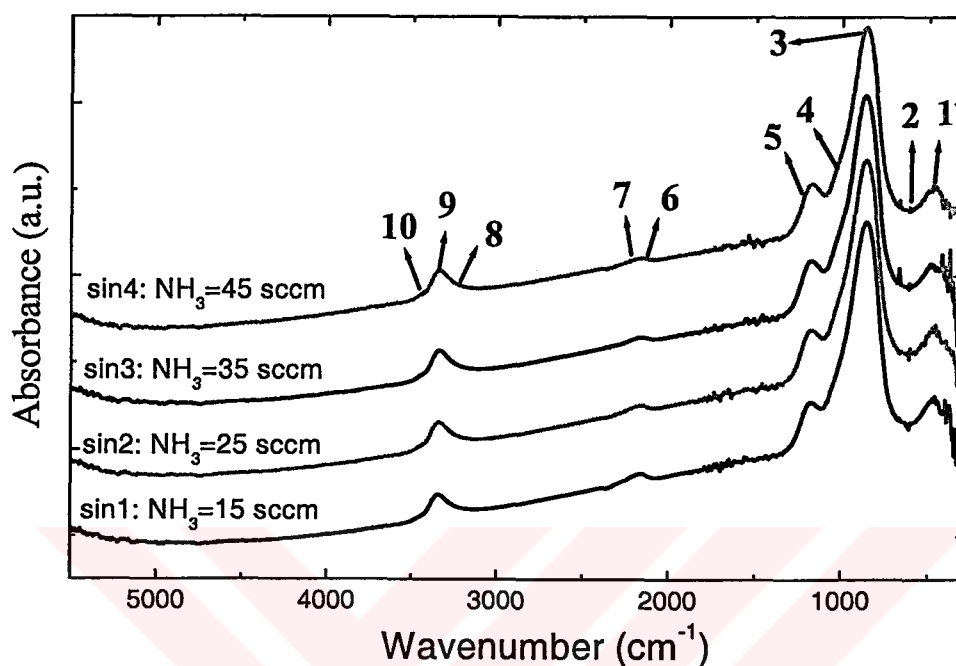


Figure 3. 9: IR absorbance of silicon nitride films grown with 15, 25, 35, and 45 sccm NH_3 flow, respectively.

Vibration Type	Peak Frequency (cm^{-1})				Reference
	sin1	sin2	sin3	sin4	
(1) Si-N breathing	474	468	472	478	49
(2) Si-H rock	665	663	673	673	49,50
(3) Si-N stretching1	843	850	857	860	49,50
(4) Si-N stretching2	957	972	996	1002	49
(5) N-H bending	1185	1184	1181	1179	49
(6) Si-H(N_2Si) stretching	2169	2158	2157	2162	48
(7) Si-H(N_3) stretching	2250	2224	2220	2235	48
(8) N-H...N stretching	3290	3293	3294	3297	41
(9) N-H stretching	3346	3345	3345	3343	40,41,48
(10) N-H ₂ stretching	3464	3462	3460	3458	48

Table 3.5: Infrared Vibrations Observed in the Grown Silicon Nitride Samples.

from Table 3.5. In Figure 3.10, all the detected infrared absorption bands for sample *sin1* are resolved into Gaussian components as was done in the previous section. The assignments of these components are as in Table 3.5.

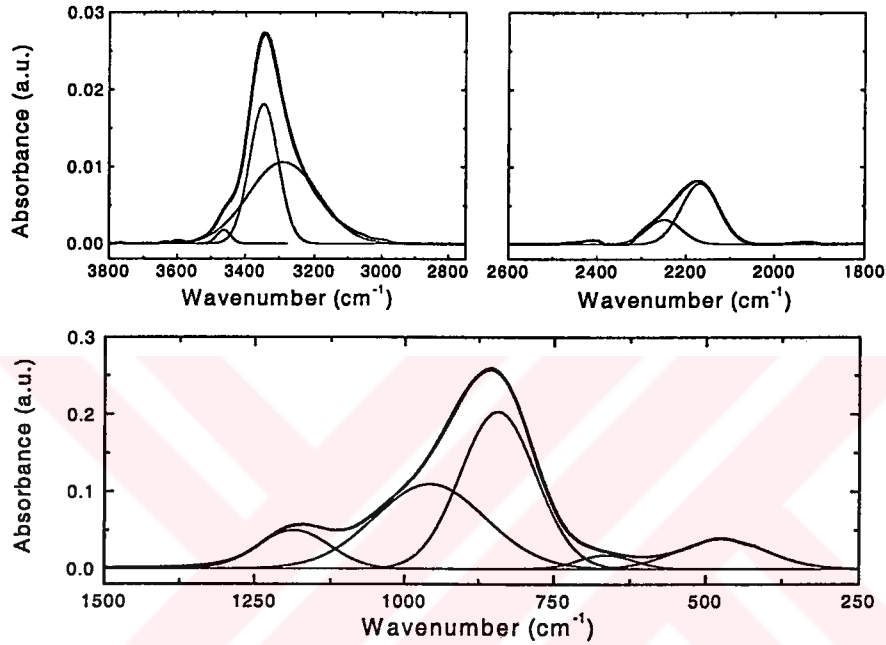


Figure 3. 10: Gaussian deconvolution of the absorption bands for sample *sin1*.

We will continue with the analysis of these bands after the bond concentrations in the films are quantified. For that purpose, we again make use of the method proposed by Lanford and Rand and the Eqn. 3.27. The absorption cross section values for N-H and Si-H bonds are given by the mentioned authors as $\sigma_{N-H} = 5.3 \times 10^{-18} \text{ cm}^2$ and $\sigma_{Si-H} = 7.4 \times 10^{-18} \text{ cm}^2$. The concentration of the Si-N bonds, on the other hand, was calculated by a cross section value in reference⁴⁵, being $\sigma_{Si-N} = 2.4 \times 10^{-17} \text{ cm}^2$. The detailed analysis of the above mentioned bond concentrations is given in Tables 3.6 and 3.7. Typical uncertainties of the involved parameters are same as in the previous section.

Sample #	Film Thickness ($\text{\AA}$)	Refractive Index	Vibration Type	Peak Wavenumber $\omega(\text{cm}^{-1})$	FWHM (cm^{-1})	Maximum Abs. Coef. $\alpha_{\text{max}}(\text{cm}^{-1})$	Normalized Abs. Band Area ($\times 10^5 \text{ cm}^{-2}$)	Sum of the Band Area (10^5 cm^{-2})	$[N - H]$ (10^{22} cm^{-3})
sin1	2861	1.85	N-H bend.	1186	139	3991	5.93		
			N-H...N str	3290	238	853	2.16		
			N-H str.	3346	99	1457	1.53		
			N-H ₂ str.	3464	47	143	0.07	9.69	7.94
sin2	2861	1.83	N-H bend.	1184	131	4274	5.96		
			N-H...N str	3293	249	958	2.54		
			N-H str.	3345	99	1562	1.65		
			N-H ₂ str.	3462	43	142	0.06	10.2	8.37
sin3	2833	1.83	N-H bend.	1181	135	4674	6.73		
			N-H...N str	3294	243	1032	2.69		
			N-H str.	3345	99	1627	1.71		
			N-H ₂ str.	3460	43	156	0.07	11.2	9.18
sin4	2753	1.81	N-H bend.	1179	137	4812	7.02		
			N-H...N str	3297	263	1029	2.89		
			N-H str.	3343	99	1648	1.75		
			N-H ₂ str.	3458	36	126	0.05	11.7	9.59

Table 3.6: N-H Bond Concentration Calculations for Silicon Nitride Films by Using FTIR Transmittance Spectroscopy.

Sample #	Film Thickness ($\text{\AA}$)	Refractive Index	Vibration Type	Peak Wavenumber $\omega(\text{cm}^{-1})$	FWHM (cm^{-1})	Maximum Abs. Coef. $\alpha_{\text{max}}(\text{cm}^{-1})$	Normalized Abs. Band Area ($\times 10^5 \text{ cm}^{-2}$)	Sum of the Band Area (10^5 cm^{-2})	$[S_i - H]$ (10^{22} cm^{-3})	$[S_i - N]$ (10^{22} cm^{-3})
sin1	2861	1.85	Si-H rock	665	106	1430	1.62			
			Si-H(N_2Si) str.	2169	100	636	0.67			
			Si-H(N_3) str.	2250	92	255	0.25	2.54	1.80	-
			Si-N br.	474	172	3107	4.35			
			Si-N str.1	843	148	16341	25.71			
sin2	2861	1.83	Si-N str.2	957	220	8854	20.64	50.70	-	9.23
			Si-H rock	663	155	1321	2.17			
			Si-H(N_2Si) str.	2158	90	359	0.34			
			Si-H(N_3) str.	2224	125	284	0.38	2.89	2.05	-
			Si-N br.	467	146	3010	3.16			
sin3	2833	1.83	Si-N str.1	850	147	17034	26.67			
			Si-N str.2	972	203	7598	16.43	46.26	-	8.42
			Si-H rock	673	105	1390	1.55			
			Si-H(N_2Si) str.	2157	87	351	0.33			
			Si-H(N_3) str.	2219	82	190	0.16	2.04	1.45	-
sin4	2753	1.81	Si-N br.	472	177	2687	2.78			
			Si-N str.1	857	153	18925	30.77			
			Si-N str.2	996	165	6324	11.08	44.63	-	8.12
			Si-H rock	673	130	1398	1.94			
			Si-H(N_2Si) str.	2162	92	320	0.31			
			Si-H(N_3) str.	2235	92	169	0.17	2.42	1.71	-
			Si-N br.	478	152	2915	4.71			
			Si-N str.1	860	153	19006	30.87			
			Si-N str.2	1001	152	5937	9.62	45.20	-	8.23

Table 3.7: Si-H and Si-N Bond Concentration Calculations for Silicon Nitride Films by Using FTIR Transmittance Spectroscopy.

The results of the calculations indicate that the N-H bond concentration is steadily increasing from $7.94 \times 10^{22} \text{ cm}^{-3}$ to $9.59 \times 10^{22} \text{ cm}^{-3}$ with the corresponding increase of the NH_3 gas flow rate. To monitor the all hydrogen concentration change in the films, the Si-H bond should be taken under consideration as well. Moreover, calculation of the concentration of species other than hydrogen should provide us with an additional information about the structure of the film. Therefore, by using the respective valencies of N and H, and assuming that there are no N-N and H-H bonds present in the layers, we relate the atomic concentration to the bond concentration in the following way,

$$[H] = [N - H] + [Si - H] \quad (3. 30)$$

$$[N] = \frac{[N - H] + [Si - N]}{3} \quad (3. 31)$$

The results of these calculations for the silicon nitride samples are given in the table below

Sample #	[H] $\times 10^{22} \text{ cm}^{-3}$	[N] $\times 10^{22} \text{ cm}^{-3}$
sin1	9.74	5.72
sin2	10.42	5.60
sin3	10.63	5.77
sin4	11.30	5.94

Table 3.8: Variation of the hydrogen and nitrogen concentrations for the samples *sin1-sin4*.

As expected, the total hydrogen concentration in the samples has increased steadily. The fact that its amount looks to be greater even from that of nitrogen, means that it can not be treated as an impurity.

In the band of interest for us (N-H stretching), hydrogen's large concentration has important impacts. Namely, as the number of N-H bonds in the layers increases, the contributions from N-H $\cdots$ N vibrational absorption increases as well. This bonding type, as proposed by *Yin and Smith*,⁴¹ takes place between the hydrogen atoms in the N-H bonds and lone pair electrons of nearby N atoms.

We explain the features of our spectra with the following arguments. The hydrogen bonding, formed between N and H, influences the characteristics of N-H

bond in a way that the original stretching vibration shifts to lower wavenumbers and becomes much broader. For our samples (see for example Fig. 2.19) the frequency difference between the N-H and N-H...N stretching modes is about $\sim 50 \text{ cm}^{-1}$ and difference in the FWHM is in the order of $\sim 100 \text{ cm}^{-1}$. In addition, as the amount of hydrogen in the layers increases there is a slight shift of $\sim 7 \text{ cm}^{-1}$ towards lower frequencies and an increase in the FWHM value of about $\sim 25 \text{ cm}^{-1}$.

The effect of the above described hydrogen bonding should also be observable in the N-H bending vibrations. Nonetheless, the corresponding peak in the IR spectrum ($\sim 1180 \text{ cm}^{-1}$) was not decomposed as to include an additional peak due to this modification. The analysis, therefore, is done by using only this band. As the concentration of hydrogen increases, we would expect the frequency of N-H bending vibration to increase. Since now hydrogen is more constrained, the needed energy for its excitation is increased, resulting in frequency increase. However, in our samples this effect is not clearly observed. The shift seems to be in the reverse direction from 1186 cm^{-1} to 1179 cm^{-1} , but this value is within the error bar, therefore arguing that there is no observable shift would be right. Instead, what we should do is to compare the frequency of the N-H bending vibration of our samples with that of the samples grown in equivalent conditions (CVD type, RF power, precursor gases used, etc.), but which contain less hydrogen. For this, we use data of Yin and Smith,⁴¹ who report values of 1163 cm^{-1} and 1181 cm^{-1} for the N-H bending mode in the samples with hydrogen of $1.77 \times 10^{22} \text{ cm}^{-3}$ and $4.24 \times 10^{22} \text{ cm}^{-3}$, respectively. The hydrogen content of these layers is about a factor of two less than ours. Thereby, it is safe to say that the amount of the frequency shift of this bands reaches a saturation point, being $\sim 1180 \text{ cm}^{-1}$, as clearly observed for our samples.

Finally, the structural study of silicon nitride films has shown that an increase in the flow rate of NH_3 results in large concentrations of hydrogen. For the growth of silicon oxynitride layers, we should consider the complications that may arise from the high flow rate of this gaseous precursor.

3.5 Compositional and Structural Properties of the As-Grown Silicon Oxynitride Layers

Silicon oxynitride samples, used in FTIR transmittance characterizations were deposited at 350^o C, an RF power of 10 W, constant 2% SiH₄/N₂ flow rate of 180 sccm, and process pressure of 1 Torr. Four samples, *sin1-sin4*, deposited with constant NH₃ flow rate of 15 sccm and N₂O flow rates of 100, 225, 300, and 450 sccm, were studied. The grown film thicknesses were about ~ 4500 Å and had index of refraction values between 1.54 and 1.48. The FTIR transmittance measurements were done in a similar manner as with silicon oxide and nitride films with the specifications given in the table below

Scanned Range	6000-250 cm^{-1}
Resolution	8 cm^{-1}
Scan Number	1024
DTGS Detector Gain	B
Interferogram Alignment Ratio	53 %
Substrate Material	Si double side polished (100)

Table 3.9: Experimental parameters of the FTIR transmittance measurements for silicon oxynitride layers.

The absorbance spectrum of the samples is depicted in Figure 3.11 with the characteristic absorption bands enumerated and identified in Table 3.10.

In the obtained infrared spectra the dominant feature is that of Si-O stretching vibration at ~ 1040 cm^{-1} , which resembles the features of silicon oxide films. The Si-O rocking and bending vibrations are detected at ~ 445 cm^{-1} and ~ 815 cm^{-1} , respectively, which is again exactly the same position as in the silicon oxide samples.

Moreover, the Si-N stretching vibration was observable only for the samples *sin1* and *sin2*. For the other samples, although there might be a certain amount of this bond present in the structure, it is not possible to decompose the band in a way to include this vibration. Most probably, as the N₂O flow rate is increased, the bonding of silicon with oxides is enhanced and the remaining Si-N bonds'

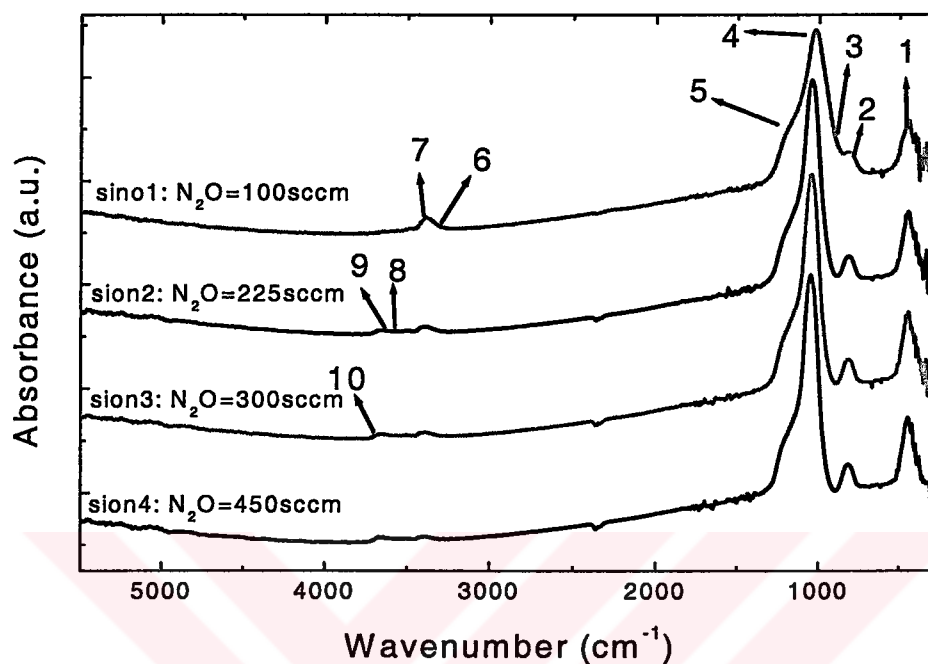


Figure 3. 11: IR absorbance of silicon oxynitride films grown with 15sccm NH_3 flow rate and N_2O flow rates of 100, 225, 300, and 450 sccm , respectively.

Vibration Type	Peak Frequency (cm^{-1})			
	sion1	sion2	sion3	sion4
(1) Si-O rock	449	445	446	443
(2) Si-O bend	815	817	816	817
(3) Si-N stretch	923	983	-	-
(4) Si-O sym. stretch	1018	1042	1040	1044
(5) Si-O asym. stretch	1154	1144	1130	1167
(6) N-H...N stretch	3341	3345	3351	3358
(7) N-H stretch	3389	3396	3399	3403
(8) H-O-H stretch	3493	3499	3499	3499
(9) SiO-H stretch1	3571	3578	3589	3589
(10)	3651	3666	3668	3670

Table 3.10: Infrared vibrations observed in the grown silicon oxynitride samples.

absorption is just obscured by it.

As for the N-H absorption band, its evolution for the four samples together with the decomposed components is given in Figure 3.12. It is clearly seen that for the samples with higher oxygen flow rate the N-H bond absorption has decreased, while the number of O-H bonds has increased.

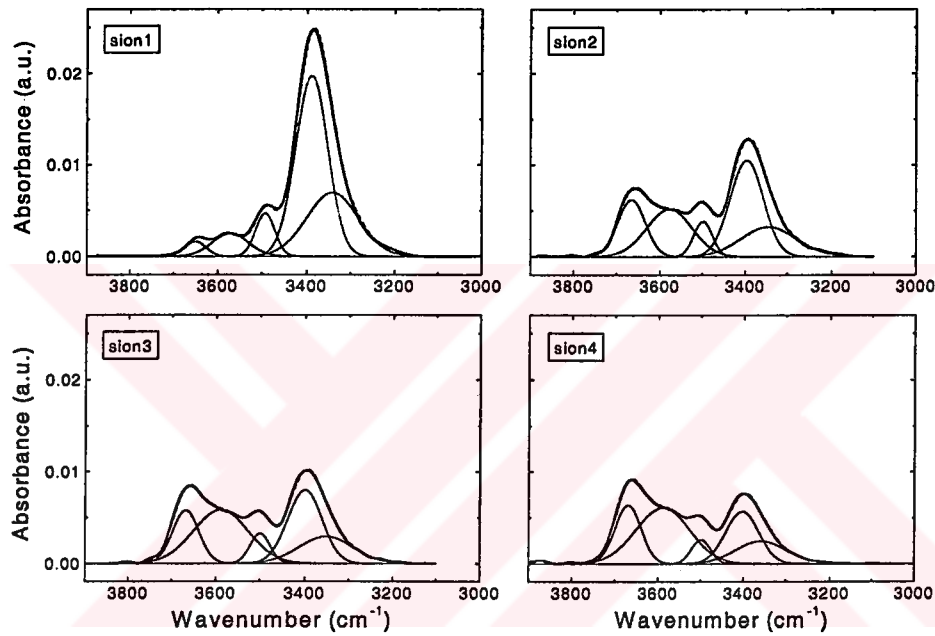


Figure 3. 12: Gaussian deconvolution of the O-H and N-H absorption bands for the samples sion1-sion4.

In order to make the compositional analysis of the samples more quantitative, the method of Lanford & Rand⁴³ is employed once again. The cross-section values for the N-H and Si-N bonds are same as the ones used for silicon nitride samples and the corresponding value for the O-H bonds is identical with the one used for silicon oxide films. Typical uncertainty values of the involved parameters are as in the section 3.3. The detailed work and its results are given in Tables 3.11 and 3.12.

First analysis that we do is to compare the bond concentrations for silicon oxynitride films with the counterparts for silicon oxide and nitride layers.

Sample #	Film Thickness ($\text{\AA}$)	Refractive Index	Vibration Type	Peak Wavenumber $\omega(\text{cm}^{-1})$	FWHM (cm^{-1})	Maximum Abs. Coef. $\alpha_{max}(\text{cm}^{-1})$	Normalized Abs. Band Area ($\times 10^5 \text{ cm}^{-2}$)	Sum of the Band Area (10^5 cm^{-2})	$[N - H]$ (10^{22} cm^{-3})	$[O - H]$ (10^{22} cm^{-3})
sion1	4409	1.54	N-H...N str.	3341	137	363	0.53	1.47	1.20	-
			N-H str	3389	86	1032	0.94			
			H-O-H str.	3493	50	244	0.13			
			SiO-H str.	3571	95	129	0.13			
				3651	55	83	0.05			
sion2	4739	1.49	N-H...N str.	3345	152	157	0.25	0.31	-	0.63
			N-H str	3396	92	510	0.50			
			H-O-H str.	3499	51	187	0.10			
			SiO-H str.	3578	124	251	0.33			
				3666	74	301	0.24			
sion3	4702	1.50	N-H...N str.	3351	142	144	0.22	0.67	-	1.54
			N-H str	3399	91	391	0.38			
			H-O-H str.	3499	52	159	0.09			
			SiO-H str.	3589	146	286	0.44			
				3668	70	284	0.21			
sion4	4638	1.48	N-H...N str.	3358	139	122	0.18	0.74	-	1.52
			N-H str	3403	91	283	0.27			
			H-O-H str.	3499	53	130	0.07			
			SiO-H str.	3589	147	303	0.47			
				3670	70	318	0.24			

Table 3.11: N-H and O-H Bond Concentration Calculations for Silicon Oxynitride Films by Using FTIR Transmittance Spectroscopy.

Sample #	Film Thickness ($\text{\AA}$)	Refractive Index	Vibration Type	Peak Wavenumber $\omega(\text{cm}^{-1})$	FWHM (cm^{-1})	Maximum Abs. Coef. $\alpha_{max}(\text{cm}^{-1})$	Normalized Abs. Band Area $(\times 10^5 \text{cm}^{-2})$	Sum of the Band Area (10^5cm^{-2})
sion1	4409	1.54	Si-O rock	449	92	4503	3.36	
			Si-O bend	815	91	2100	2.03	
			Si-O sstr.	1018	127	13215	17.92	
			Si-O as.str.	1154	180	5955	11.38	34.7
			Si-N str.	923	100	2794	2.96	2.96
sion2	4739	1.49	Si-O rock	445	80	5430	4.10	
			Si-O bend	817	76	2250	1.81	
			Si-O str.	1042	93	14866	14.66	
			Si-O as.str.	1144	198	6908	14.56	35.1
			Si-N str.	983	99	2935	3.10	3.10
sion3	4702	1.50	Si-O rock	446	78	5745	3.99	
			Si-O bend	816	70	2336	1.73	
			Si-O str.	1040	95	15585	15.78	
			Si-O as.str.	1130	227	7180	17.35	38.8
sion4	4638	1.48	Si-O rock	443	79	5829	4.30	
			Si-O bend	817	69	2343	1.71	
			Si-O str.	1044	101	19266	20.68	
			Si-O as.str.	1167	170	6341	11.47	38.2

Table 3.12: Si-N and Si-O Bond Concentration Calculations for Silicon Oxynitride Films by Using FTIR Transmittance Spectroscopy.

Beginning with the critical bonding type, N-H, in silicon oxide samples its concentration varied between $0.74 \times 10^{22} \text{ cm}^{-3}$ and $0.04 \times 10^{22} \text{ cm}^{-3}$, with corresponding N_2O flow rate ranging between 25 and 300 sccm. In silicon nitrides the N-H bond concentration was found to be $(7.94\text{-}11.7) \times 10^{22} \text{ cm}^{-3}$, more than a factor of 9 greater than in silicon oxide layers, with NH_3 flow rate of 15-45 sccm. Now, in silicon oxynitride layers, which are grown with constant NH_3 flow of 15 sccm and varying flow of N_2O flow rate of 100-450 sccm, the mentioned bond concentration ranged between $1.20 \times 10^{22} \text{ cm}^{-3}$ and $0.37 \times 10^{22} \text{ cm}^{-3}$. The comparison of the N-H bond concentration variation with N_2O flow rate for silicon oxynitride and oxide samples is given below in Figure 3.13. We observe that for both type of the films there is a decrease in the N-H bond concentration for more than three times. This can be understood in a way that, as the oxygen concentration in the layers increases it begins to dominate over the bonds formed with hydrogen, due to being more electronegative than nitrogen. Moreover, from

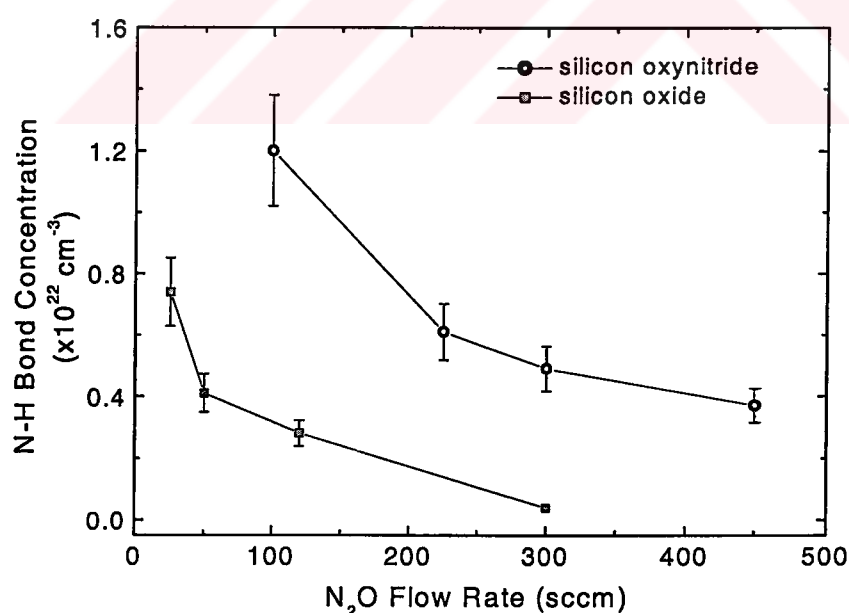


Figure 3. 13: N-H bond concentration variation with N_2O flow rate for silicon oxynitride and silicon oxide films.

the Table 3.11 we see that for both types of films there is a trend of increase in the number of O-H bonds as N_2O flow rate is increased, which further supports our arguments.

In addition, if we specifically monitor the N-H...N bonding related absorption, we observe a decreasing concentration, as well. This is due to the fact that now hydrogen forms bonds mainly with oxygen, resulting in less N-H and thus N-H...N bonds, being consistent with our results. As for the other bond types, from Table 3.12 we see that the number of the Si-O bonds increases steadily with increasing N_2O flow, which is what we would expect. Here, it should be noted that the Si-O bonds seem to dominate over Si-N ones, which again can be explained by the previous arguments.

The variation of the total hydrogen concentration for silicon oxynitride films can be calculated using the relation

$$[H] = [N - H] + [O - H] \quad (3.32)$$

the results of which are tabulated below

Sample #	[H] $\times 10^{22} cm^{-3}$
sion1	1.83
sion2	2.15
sion3	2.01
sion4	2.03

Table 3.13: Variation of the hydrogen concentration for the samples *sion1-sion4*.

The uncertainty of the above values are as discussed in section 3.3. The hydrogen concentration of silicon oxide sample *o3*, grown with the same N_2O flow of 300 sccm as *sion3*, is calculated by using Eqn. 3.32, too. The value found is $1.47 \times 10^{22} cm^{-3}$, which shows that the corresponding silicon oxynitride film contains about % 54 more hydrogen. The other extreme is the sample *sin1*, which was grown with NH_3 flow of 15 sccm as was done in samples *sion1-sion4*. Its hydrogen concentration is $9.74 \times 10^{22} cm^{-3}$ being 6.6 times more than that of sample *o3* and 4.5 times more than sample *sion3*.

The performed infrared study on silicon oxide, nitride, and oxynitride films has proven to be effective method for compositional analysis of the grown layers. As it was aimed, the conditions affecting the hydrogen incorporation into the films were identified. In particular, the grown silicon oxynitride films were shown to have more resemblance with the oxide layers than nitride films, in terms of both types of detected vibrations and their amount in the structure.



3.6 Annealing Study

At this step of the study we have to choose a specific silicon oxynitride layer, planned to serve as the core layer in an optical waveguide. The structure of the waveguide will be as follows. On a silicon substrate there will be $\sim 7 \mu\text{m}$ thick SiO_2 buffer layer, on which the SiON film will be grown. In addition, the whole structure will be covered with SiO_x layer grown by PECVD. In choosing the specific oxynitride film type two factors are to be considered. The decided index of contrast between the core and the cladding layers introduces the first restriction. And the other parameter is the amount of N-H bond present in the silicon oxynitride layer. Considering the previous work done on refractive index and N-H bond concentration characterizations (sections 2.4 & 3.5), we choose a silicon oxynitride film of refractive index of ~ 1.50 , corresponding to flow rates of NH_3 and N_2O of 15 sccm and 225 sccm, respectively (sample sion2).

This sample still contains certain amount of N-H bonds, that are known to be the main cause of optical absorption in the waveguides. Therefore, we devote the rest of this chapter to a study performed in order to decrease or eliminate this type of bonding from the film structure by an annealing treatment. Below, the annealing furnace setup is described. After which, the results of the FTIR analysis of the annealed samples are discussed and a concluding study using attenuated total reflection (ATR) infrared spectroscopy is worked out.

3.6.1 Annealing Furnace

The hydrogen, incorporated into the silicon oxynitride layers, in general is desorbed by applying a heat treatment. For our case, the annealing experiments were done using a commercial *Protherm* furnace, capable of maximum heating temperature of 1350°C . The samples to be annealed were placed on a quartz boat inside an alsint tube of 110 cm length and diameter of 5 cm. For the experiments to be repeatable, a constant ambient of nitrogen was setup, the flow rate of which was held fixed at 7 lt/min. Because of this, water cooled caps were attached on the both ends of the tube. The schematics of the used annealing furnace is given

in Figure 3.14.

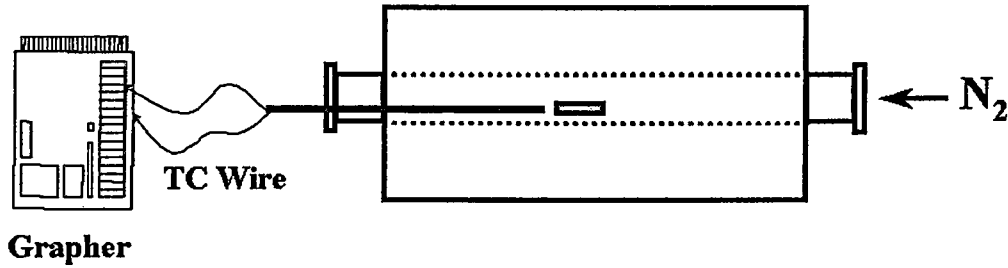


Figure 3. 14: Annealing furnace setup.

During the experiments, the temperature in the neighborhood of the sample was monitored using a chromel-alumel thermocouple (TC) wire, connected to an analog grapher. A temperature controller built-in the furnace was employed in order to program the temperature variations during the annealing experiments. Moreover, an additional measurement was performed along the tube length, so as to determine the extent of the region of constant temperature in the tube. At a temperature set to 1350 °C the measured values are plotted in Figure 3.15. As it is seen, the range of about ~10 cm at the centre of the tube is at constant temperature and is suitable for our applications.

3.6.2 FTIR Analysis of the Annealed Samples

The characteristics of the sample *sion2* (see section 3.5) were chosen to fit our requirements as the core layer material for optical waveguides. Therefore, silicon oxynitride films grown with 225 sccm N₂O and 15 sccm NH₃ flow rates and having refractive index of ~1.50 are worked on in the annealing studies. The film thicknesses were of about ~4500 Å and were deposited on double side polished Si(100) substrates.

In order to observe the change in the N-H bond concentration in the layers, four different annealing programs were run. The programs had equal ramping rates between 0-700 °C and 700- T_{max} with two hours of annealing at maximum

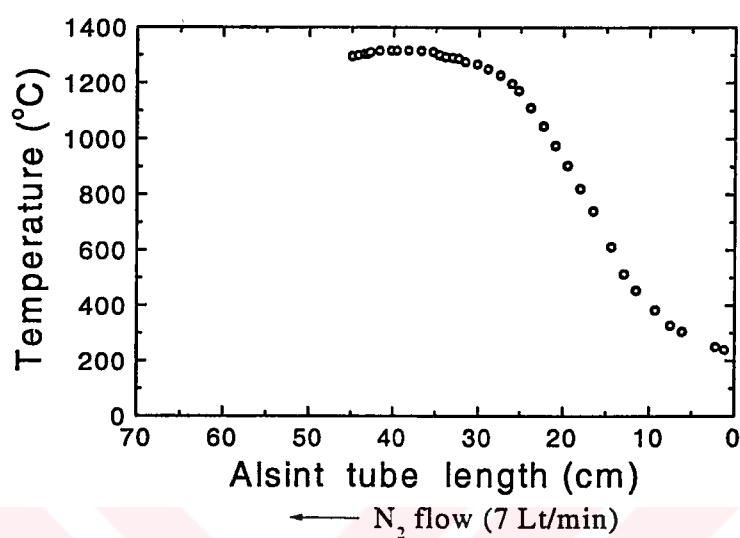


Figure 3. 15: Temperature profile along the annealing furnace tube at maximum temperature set to 1350°C.

temperature. Initially four programs at temperatures of 800 °C, 900 °C, 1000 °C, and 1100 °C were applied. A sample annealing program is given in Figure 3.16.

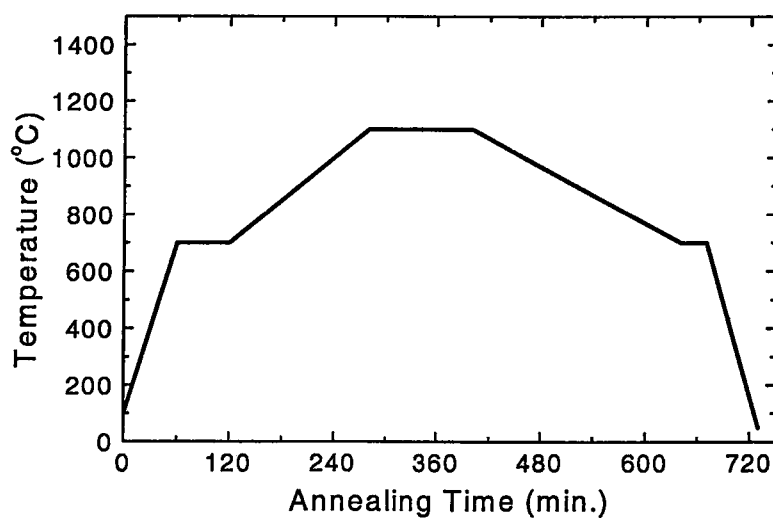


Figure 3. 16: Sample annealing program for two hours at 1100°C.

The samples studied were deposited at identical conditions as mentioned above and given in Table 2.4. Their FTIR transmittance measurements were performed as previously described in Table 3.9. The absorbance spectrum of the layers is depicted in Figure 3.17, and their identification of absorption bands in Table 3.14.

The annealing treatment has some striking effects on the infrared spectra and thus on the structure of the films. We observe a definite trend of narrowing of the bands, which means that the extent of different atomic arrangements surrounding the bonds has decreased. This, in turn, implies that the structure of the layer has become more ordered. In addition, a strong shift of the Si-O-Si stretching frequency is evidenced. It is attributed to the shortening of the average bond lengths leading to an increase in the average vibrational frequency. The process is accompanied by the densification of the film by release of atoms from it, which leads to a clearly observed tensile stress in our structures. Moreover, the increase in the stretching frequency of the Si-O-Si stretching vibration up to 1079 cm^{-1} means that the Si-O-Si angle increases to the value corresponding to that of thermally grown silicon oxide layers.

The most important feature of the spectra, as far as we are concerned, is the strong reduction of the vibrations related to hydrogen. In order to relate its concentration in the films to the annealing temperature, we make an analysis

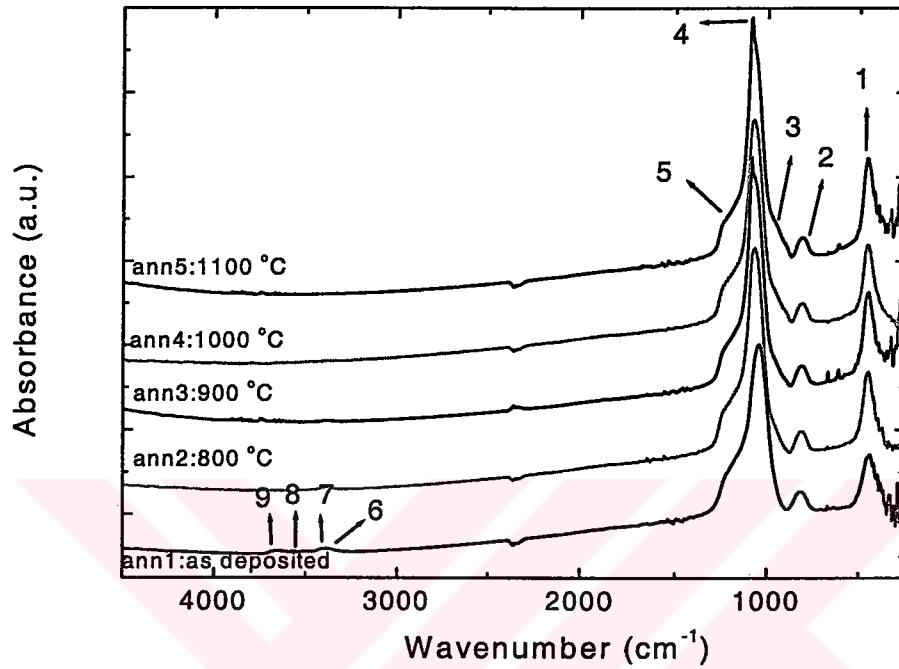


Figure 3. 17: IR absorbance spectra of silicon oxynitride films annealed at 800 °C, 900 °C, 1000 °C, and 1100 °C .

Vibration Type	Peak Frequency (cm^{-1})				
	ann1	ann2	ann3	ann4	ann5
(1) Si-O rock	442	452	451	453	454
(2) Si-O bend	817	812	812	809	808
(3) Si-N stretch	980	971	1024	988	1034
(4) Si-O s.stretch	1044	1065	1073	1071	1079
(5) Si-O as.stretch	1153	1182	1189	1185	1196
(6) N-H...N stretch	3344	-	-	-	-
(7) N-H stretch	3399	3389	3386	-	-
(8) H-O-H stretch	3501	-	-	-	-
(9) SiO-H stretch1	3582	-	-	-	-
(10)	3666	-	-	-	-

Table 3.14: Infrared vibrations observed in the annealed samples.

similar to the ones done for oxide, nitride, and oxynitride layers previously. Again, the method of Lanford & Rand is employed and the results are tabulated in Table 3.17.

The evolution of the N-H stretching band with the annealing temperature is given in Figure 3.18 in a more closer view.

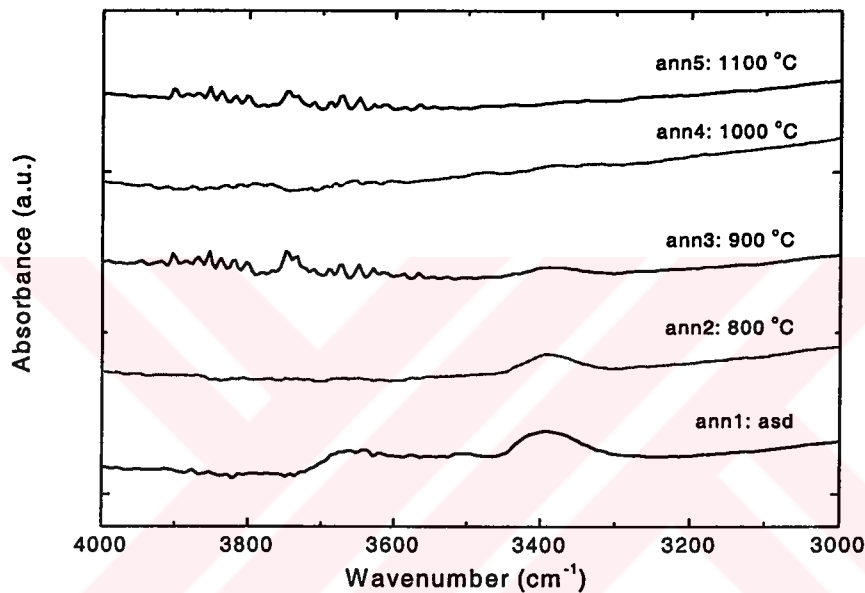


Figure 3. 18: Evolution of the N-H stretching band with the annealing temperature.

It is obvious that the O-H related absorption bands are eliminated upon first annealing at 800 °C, while the N-H stretching vibration is still detectable. Nevertheless, with an increase in the annealing temperature the area of this band also decreases and goes below the detection limit at temperature of 1000 °C. The quantitative variation of the N-H bond concentration with the annealing temperature is given in Figure 3.19. The total hydrogen concentration in the as deposited films is expected to be slightly more than the value given in Table 3.15, because the N-H bending vibration is obscured due to Si-O stretching absorption at $\sim 1150\text{cm}^{-1}$. However, if we analyze the corresponding bands of the sample ann5 (annealed at 1100 °C), in which we expect no N-H bonds, we see that there

Sample #	Film Thickness ($\text{\AA}$)	Refractive Index	Annealing Temperature $^{\circ}\text{C}$	Vibration Type	Peak Wavenumber $\omega(\text{cm}^{-1})$	FWHM (cm^{-1})	Maximum Abs. Coef. $\alpha_{\text{max}}(\text{cm}^{-1})$	Normalized Abs. Band Area ($\times 10^5 \text{ cm}^{-2}$)	Sum of the Band Area (10^5 cm^{-2})	[N - H] (10^{22} cm^{-3})
ann1	4703	1.48	asd	N-H...N str.	3344	131	135	0.19	0.64	0.52
				N-H str	3399	93	450	0.45		
				H-O-H str.	3501	54	160	0.09		
				SiO-H str.(1)	3582	123	250	0.33		
				SiO-H str.(2)	3666	70	283	0.21		
ann2	4718	1.48	800	N-H str.	3389	81	284	0.25	0.25	0.20
ann3	4596	1.48	900	N-H str.	3386	77	137	0.11	0.11	0.09
ann4	4589	1.49	1000	N-H str.	-	-	-	-	-	-
ann5	4563	1.49	1100	N-H str.	-	-	-	-	-	-

Table 3.15: N-H and O-H bond concentration calculations for the annealed silicon oxynitride films by using FTIR transmittance spectroscopy.

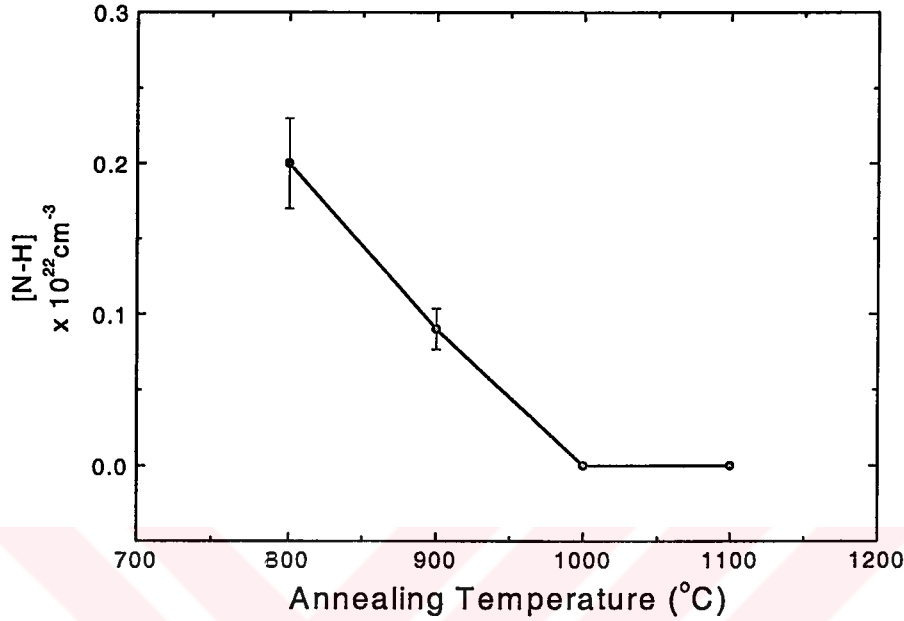


Figure 3. 19: Evolution of the N-H bond stretching concentration with the annealing temperature.

is no considerable difference, indicating that the contribution of N-H bending to be very small. Concentrating on the N-H stretching bond concentration, we observe that it decreases from $0.52 \times 10^{22} \text{ cm}^{-3}$ for the as-deposited sample ann1, to $0.09 \times 10^{22} \text{ cm}^{-3}$ for the sample ann3 annealed at 900°C and goes below detection limit after 1000°C . Thus, according to the performed infrared analysis, we achieve our aim of eliminating the N-H bond stretching at an annealing temperature of 1000°C .

The refractive index and thickness measurements of the annealed samples gave interesting results as well. The corresponding values were measured by an ellipsometer at 632.8 nm wavelength. The results are tabulated in the Table 3.16. The uncertainty in the index of refraction is about ± 0.01 and in the thickness measurement $\pm 10 \text{ \AA}$, however in the table the indicated values for the thickness correspond to its variation over a 1 cm^2 area in the centre of the samples. The as deposited and annealed film thicknesses of the samples of consideration are

Sample #	Ann. Temp. ($^{\circ}\text{C}$)	n_{asd}	t_{asd} ($\text{\AA}$)	n_{ann}	t_{ann} ($\text{\AA}$)
ann2	800	1.49	4730 ± 40	1.48	4718 ± 20
ann3	900	1.49	4703 ± 40	1.48	4596 ± 10
ann4	1000	1.48	4808 ± 44	1.49	4590 ± 8
ann5	1100	1.49	4796 ± 60	1.49	4563 ± 10
ann6	1050	1.48	4818 ± 36	1.48	4538 ± 8
ann7	1150	1.48	4842 ± 77	1.49	4530 ± 11
ann8	1200	1.48	4740 ± 25	1.49	4405 ± 5

Table 3.16: Refractive index and thickness measurements of the annealed samples.

plotted in Figure 3.20 where their variations are indicated as error bars. In the

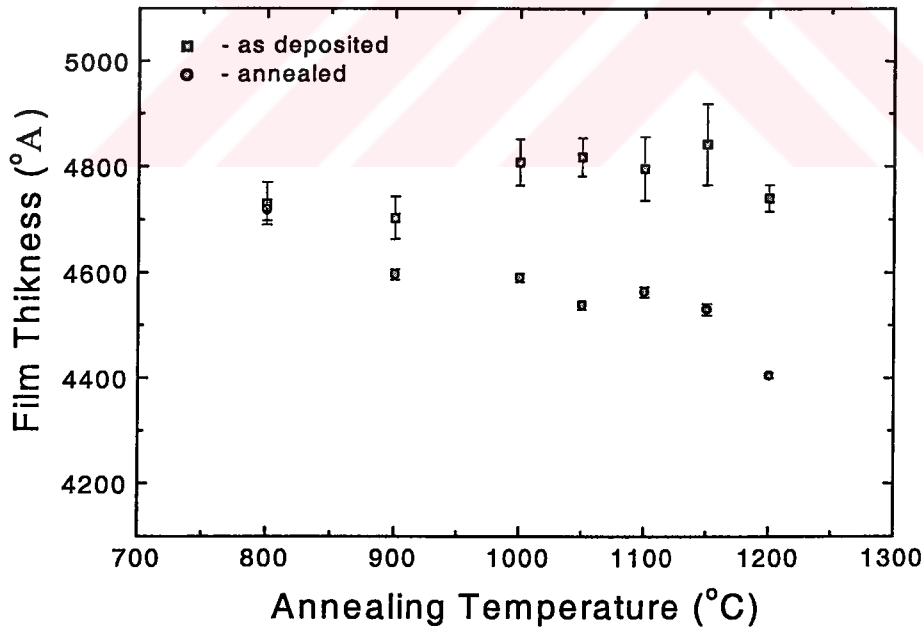


Figure 3. 20: Thickness measurements of the as deposited and annealed samples.

layers there was no measurable change of the refractive index. However, the thicknesses of the films has decreased by 2 % for 900 °C annealing and by 7 % for 1200 °C annealing. This can be attributed to the densification of the network and probably to release of the gaseous NH species.

3.6.3 ATR Technique

Having determined that no N-H bonds are present in the silicon oxynitride layers after annealing at 1000 °C, we would like to confirm this with a more sensitive method than infrared transmission. The attenuated total reflection (ATR) technique provides us with the necessary accuracy. For this purpose, we have used a silicon ATR parallelogram crystal of 45° with dimensions 5 mm × 3 mm × 50 mm (see Figure 3.21). The structure to be analyzed was grown on the crystal, after which it was annealed and FTIR ATR spectrum taken by using a special attachment.

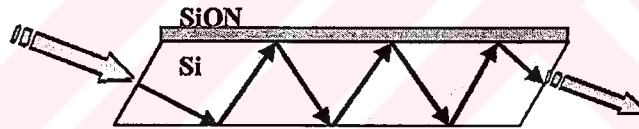


Figure 3. 21: ATR crystal used for N-H bond detection.

The advantage of this technique comes from the multiple internal reflections that take place in the crystal. That is, as the light is reflected from the SiON/Si interface, its amplitude decreases in the direction of the film. When the wave penetrates the oxynitride layer by an amount

$$P.D. = \frac{\lambda}{2\pi \sqrt{\left(\frac{\sin^2 \theta}{(n_{SiON}/n_{Si})^2} - 1\right)}} \quad (3. 33)$$

where θ is the angle of incidence, its amplitude is decreased by a factor of $1/e$. The energy of this evanescent wave returns to the crystal unless another medium is introduced above SiON. Moreover, the number of reflections is equal to

$$N = \frac{L \cot \theta}{d} \quad (3. 34)$$

where L and d are the length and thickness of the crystal, respectively. Thus, for our case the total number of reflections is equal to ~ 16 and the penetration depth profile is as in Figure 3.22

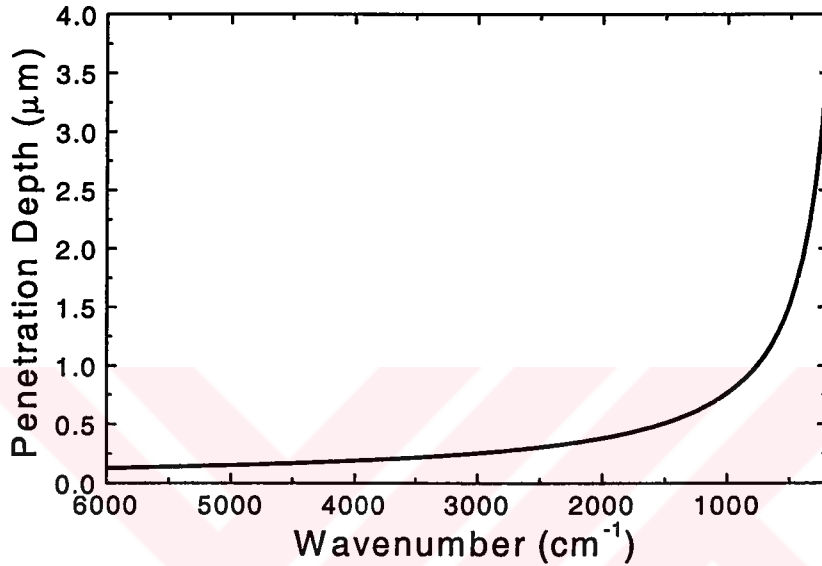


Figure 3. 22: Penetration depth profile for the silicon - silicon oxynitride structure.

At the frequency of N-H stretching, i.e. $\sim 3500 \text{ cm}^{-1}$, light penetrates into the SiON layer only for about $0.2 \text{ } \mu\text{m}$. Therefore, the films used in our characterizations were about $\sim 0.5 \text{ } \mu\text{m}$ thick. At similar condition of the FTIR setup with the previous measurements and perpendicular incidence of the light onto inclined side of the crystal, four spectra were taken. The analyzed samples were annealed at $900 \text{ }^{\circ}\text{C}$ and $1000 \text{ }^{\circ}\text{C}$ identically as the samples ann3 and ann4. In addition, one more annealing was performed at temperature of $1150 \text{ }^{\circ}\text{C}$ but now for four hours. The graph of their spectra for the N-H stretching vibration region is given in Figure 3. 23.

Although in the spectra of Fig. 3.18 it was observed that there is no N-H stretching vibration related absorption at $1000 \text{ }^{\circ}\text{C}$ annealing, from the ATR analysis its presence is strongly evident. However, at an annealing program of 4 hours at $1150 \text{ }^{\circ}\text{C}$, the N-H bond concentration in the film may be assumed to be

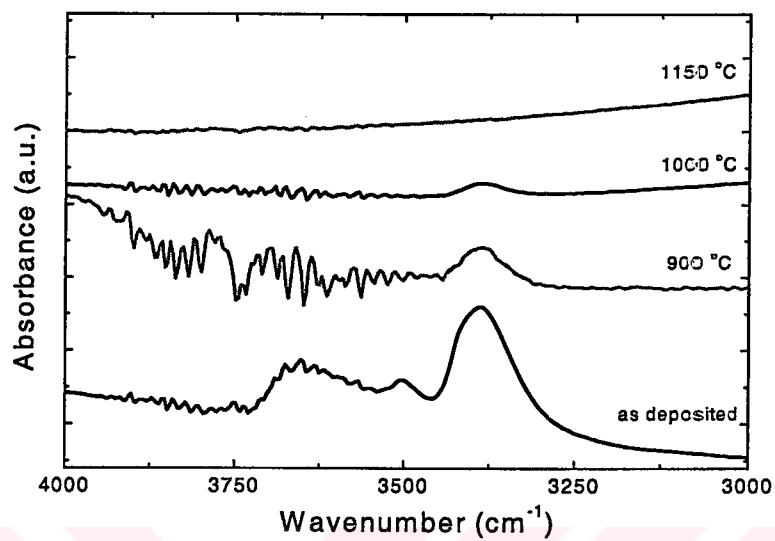


Figure 3. 23: N-H stretching vibration evolution as detected by ATR infrared spectroscopy.

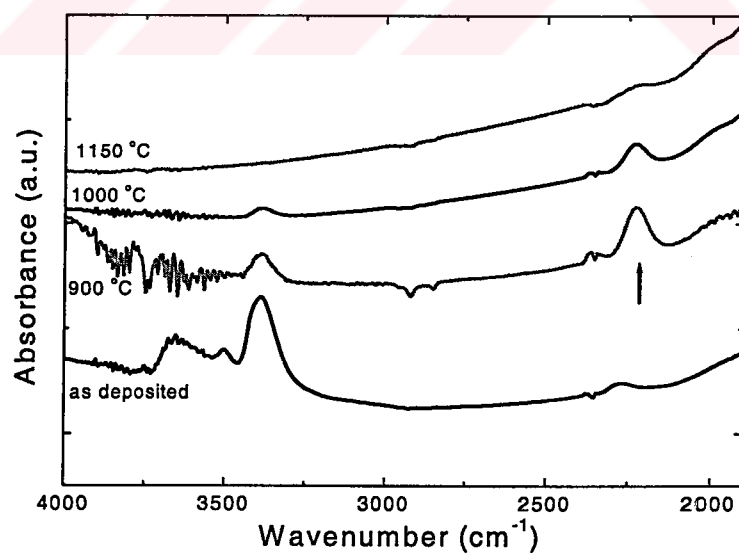


Figure 3. 24: Si-H stretching vibration evolution as detected by ATR infrared spectroscopy.

nearly zero.

Another advantage of the ATR over the conventional transmission spectra is evident from the Figure 3.24. Here, the vibration due to Si-H bond stretching at $\sim 2200\text{ cm}^{-1}$ is detected. Actually, it is definitely present in all the samples, except for the one annealed at $1150\text{ }^{\circ}\text{C}$. This vibration type was not detected in the previous study of the annealed samples. The reason for this is the low depth of penetration of the light in the ATR crystal setup, which results in a closer analysis of the Si/SiON interface itself. From the Figure 3.24 we see that after first annealing at $900\text{ }^{\circ}\text{C}$, the Si-H bond concentration seem to increase and after that begins to decrease. Here, hydrogen tends to form bonds with silicon atoms from the substrate. This formation is observed also partly because of the desorption of nitrogen itself together with hydrogen which leads to decrease in its amount. As the film is further annealed, this bond type begins to diminish too, and becomes undetectable in the sample annealed at $1150\text{ }^{\circ}\text{C}$.

In conclusion, we have verified by infrared transmission and ATR analysis that there is no observable N-H bond present in the structure of the films, after an annealing program at $1150\text{ }^{\circ}\text{C}$ for four hours.

Chapter 4

Waveguide Fabrication

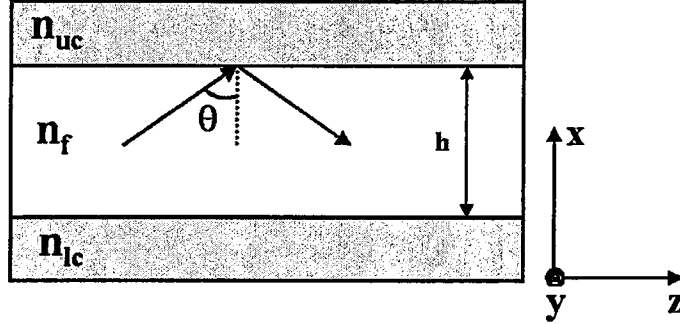
This chapter describes the process steps applied in the fabrication of silicon oxynitride based optical waveguides. The mode confinement conditions for slab and ridge waveguides are analyzed together with the possible loss mechanisms in our structures. Finally, the strip definition process for the ridge waveguides by reactive ion etching (RIE) is presented, as well as the remaining steps in the fabrication study.

4.1 Theoretical Background as Applied to SiON Based Waveguides

4.1.1 Optical Mode Confinement for a Slab Waveguide

Consider planar waveguiding structure which is formed by stacking three different layers on each other (see Figure 4.1). The layer in the middle has the highest index of refraction, because of which it confines in itself most of the field intensity due to total internal reflections. This type of waveguiding structure is called as a slab waveguide, where the upper and lower layers are called as upper and lower cladding, respectively.

The behavior of light in the above structure is derived from the Maxwell's

Figure 4. 1: Structure of a slab waveguide ($n_f > n_{lc}, n_{uc}$).

equations, which for the linear, lossless, and isotropic medium are in the form of

$$\vec{\nabla} \times \vec{E} = -\mu_0 \frac{\partial \vec{H}}{\partial t} \quad \vec{\nabla} \times \vec{H} = \epsilon_0 n^2 \frac{\partial \vec{E}}{\partial t} \quad (4. 1)$$

where n is the refractive index of the medium and μ_0 and ϵ_0 are the magnetic permeability and dielectric permittivity of free space, respectively.⁵¹ The scalar wave equation is obtained from them to give

$$\nabla^2 \psi + k_0^2 n^2 \psi = 0 \quad (4. 2)$$

where ψ stands for E or H . Choosing z as the direction of propagation, a trial solution for Eqn. 4.2 is written in the form

$$E_y(x, z) = E_y(x) e^{-i\beta z} \quad (4. 3)$$

where E_y is the electric field polarized in the y direction which is y -independent due to symmetry along that direction and β is the propagation constant along the z direction for the core, upper or lower cladding layers.

Substituting this trial solution in Eqn. 4.2 we obtain

$$\frac{\partial^2 E_y}{\partial x^2} + (k_0^2 n_i^2 - \beta^2) E_y = 0 \quad (4. 4)$$

where n_i depends on the x position where the equation is applied. This transverse wave equation has general solutions of the form

$$E_y(x) = E_0 e^{\pm \sqrt{\beta^2 - k_0^2 n_i^2} x} \quad \text{for } \beta > k_0 n_i \quad (4. 5)$$

$$E_y(x) = E_0 e^{\pm i \sqrt{k_0^2 n_i^2 - \beta^2} x} \quad \text{for } \beta < k_0 n_i \quad (4. 6)$$

which means that depending on the value of β the solution is either oscillatory or exponentially decaying. For the decaying case an attenuation coefficient is defined as

$$\gamma = \sqrt{\beta^2 - k_0^2 n_i^2} \quad (4. 7)$$

and the field is described as

$$E_y(x) = E_0 e^{-\gamma x} \quad (4. 8)$$

For the case when $\beta < k_0 n_f$ a transverse wavevector κ is defined as

$$\kappa = \sqrt{k_0^2 n_i^2 - \beta^2} \quad (4. 9)$$

such that $E_y(x)$ becomes

$$E_y(x) = E_0 e^{\pm i\kappa x} \quad (4. 10)$$

Using the condition for the guided wave to exist $k_0 n_{lc} < \beta < k_0 n_f$, the transverse portion of the electric field amplitudes in the three regions are written as

$$\begin{aligned} E_y(x) &= A e^{-\gamma_{uc} x} & \text{for } 0 < x \\ E_y(x) &= B \cos(\kappa_f x) + C \sin(\kappa_f x) & \text{for } -h < x < 0 \\ E_y(x) &= D e^{\gamma_{lc}(x+h)} & \text{for } x < -h \end{aligned} \quad (4. 11)$$

where A, B, C, and D are coefficients to be determined from the boundary conditions and $x = 0$ coordinate is chosen at the boundary at the top interface.

Applying the boundary conditions for continuity of tangential E and B at the interfaces and doing several mathematical manipulations using Eqn. 4.11, the eigenvalue equation for β is obtained, giving

$$\tan(h\kappa_f) = \frac{\gamma_{uc} + \gamma_{lc}}{\kappa_f \left[1 - \frac{\gamma_{uc}\gamma_{lc}}{\kappa_f^2} \right]} \quad (4. 12)$$

for the TE modes, and

$$\tan(h\kappa_f) = \frac{\kappa_f \left[\frac{n_f^2}{n_{lc}^2} \gamma_{lc} + \frac{n_f^2}{n_{uc}^2} \gamma_{uc} \right]}{\left[\kappa_f^2 - \frac{n_f^4}{n_{uc}^2 n_{lc}^2} \gamma_{uc} \gamma_{lc} \right]} \quad (4. 13)$$

for TM polarization.

The above equation can be used in order to determine the number of modes that a slab waveguide can support. For our case silicon oxynitride layer with refractive index of 1.50 and thickness of $2.0 \mu\text{m}$ is used as the core material. Thermally grown silicon oxide layer of $7.2 \mu\text{m}$ thickness and index of 1.46 serves as a lower cladding material and air can be assumed to be the upper cladding. Making use of Eqn. 4.11, it was estimated that the described slab waveguide supports two modes for $\lambda = 632.8 \text{ nm}$ (see Figure 4.2).

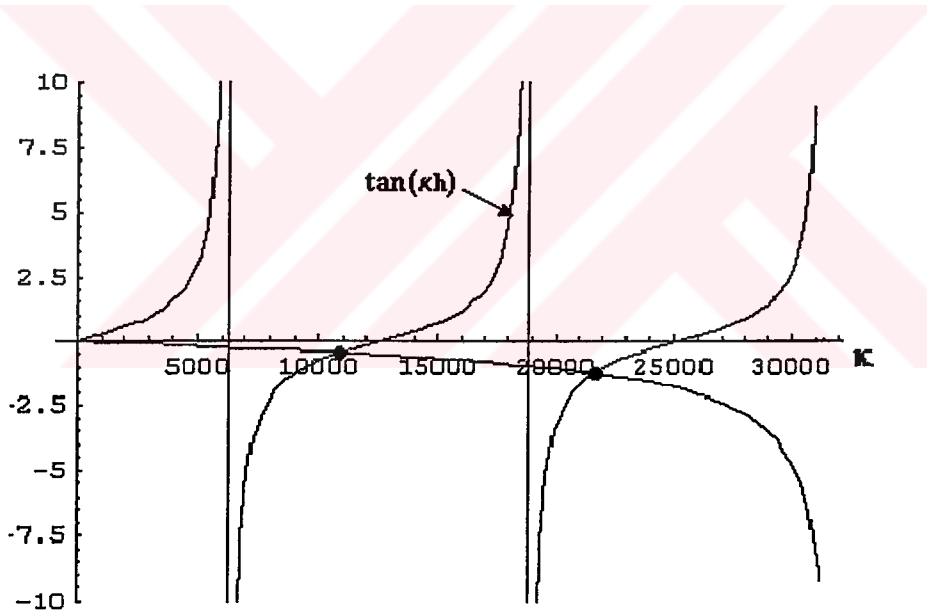


Figure 4. 2: Numerical solution of the transcendental characteristic eqn. 4.12 for the allowed κ values for SiON slab waveguide.

4.1.2 Single Mode 3D Waveguides and the Effective Index Method

The discussed planar slab waveguides are relatively easy to fabricate, however they provide confinement only in one dimension which limits its applicability. Many applications in optical integrated circuits require two dimensional optical confinement that is achieved by making use of, for example, ridge waveguides. Our aimed structure of the waveguide is an example for this and is depicted in Figure 4.3. In this structure, the index of refraction of SiON ($n_{SiON} > n_{SiO_2}$)

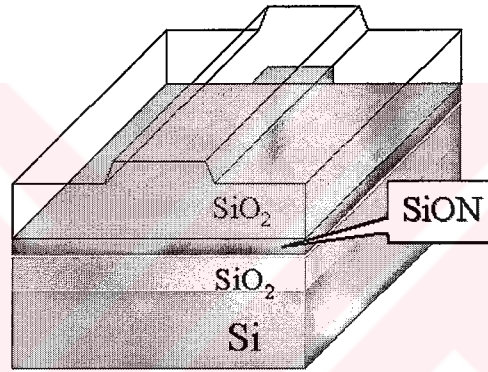


Figure 4. 3: Schematics of a ridge waveguide with SiON core layer and strip and SiO₂ upper and lower claddings on silicon substrate.

provides the vertical confinement and the rib (or strip) etched on the top layer introduces the horizontal confinement.

The approximate modal characteristics of the above structure can be calculated by using the well known *effective index method*. This method converts a two-dimensional problem into several one-dimensional problems. Considering our structure, the waveguide geometry is divided along the symmetry lines as shown in Figure 4.4. The obtained slab waveguides are analyzed for TE and TM modes in order to find the allowed values for β at a particular wavelength. Then, the effective index for the each slab is determined from the equation

$$n_{eff} = \frac{\beta}{k_0} \quad (4. 14)$$

where k_0 is the wavenumber of the light in vacuum. At each step of calculation the effective index found previously is used and the finally obtained value of β is the desired one.

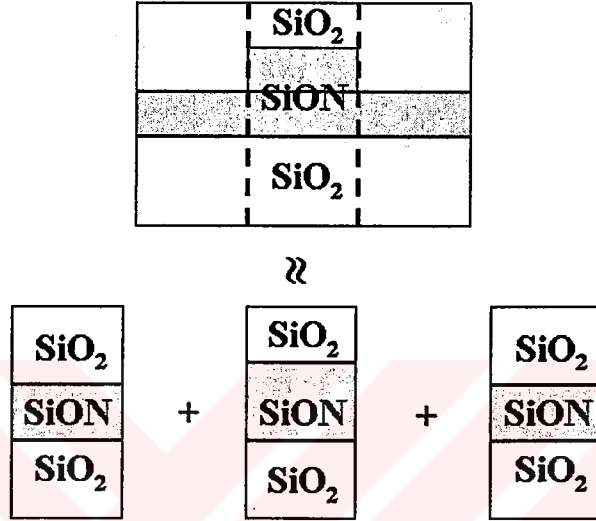


Figure 4. 4: Calculation steps in the effective index method.

Having studied the method for calculation of effective indices, the condition for single-mode guiding of our structure is analyzed. The desired 3-D waveguide is aimed to be single-mode one, i.e. to support the fundamental mode only. For this, we make use of the work based on effective index method of Pogossian et al.,⁵² which reads as

$$t < \frac{r}{\sqrt{1-r^2}} + c \quad (4. 15)$$

where

$$\begin{aligned} c &= 0.5 \\ t &= \frac{\omega_{eff}}{H_{eff}} \\ r &= \frac{h_{eff}}{H_{eff}} \\ h_{eff} &= h + q \\ H_{eff} &= H + q \end{aligned} \quad (4. 16)$$

$$\omega_{eff} = \omega + \frac{2\gamma_{uc}}{k\sqrt{n_f^2 - n_{uc}^2}}$$

$$q = \frac{\gamma_{uc}}{k\sqrt{n_f^2 - n_{uc}^2}} + \frac{\gamma_{lc}}{k\sqrt{n_f^2 - n_{lc}^2}}$$

and n_f , n_{uc} , n_{lc} are the refractive indices of the guiding region, upper and lower cladding, respectively. H is the total thickness of the slab plus the rib, h is the slab thickness of the guiding film and ω is the width of the rib.

The relations 4.13 and 4.14 were used for designing a single mode waveguide with SiON core layer of index 1.50 and lower and upper cladding layers with refractive index of 1.46 at $\lambda = 1.55 \mu\text{m}$. The results of the calculations are given graphically in Figure 4.5. Here, in the region below the line, the structure

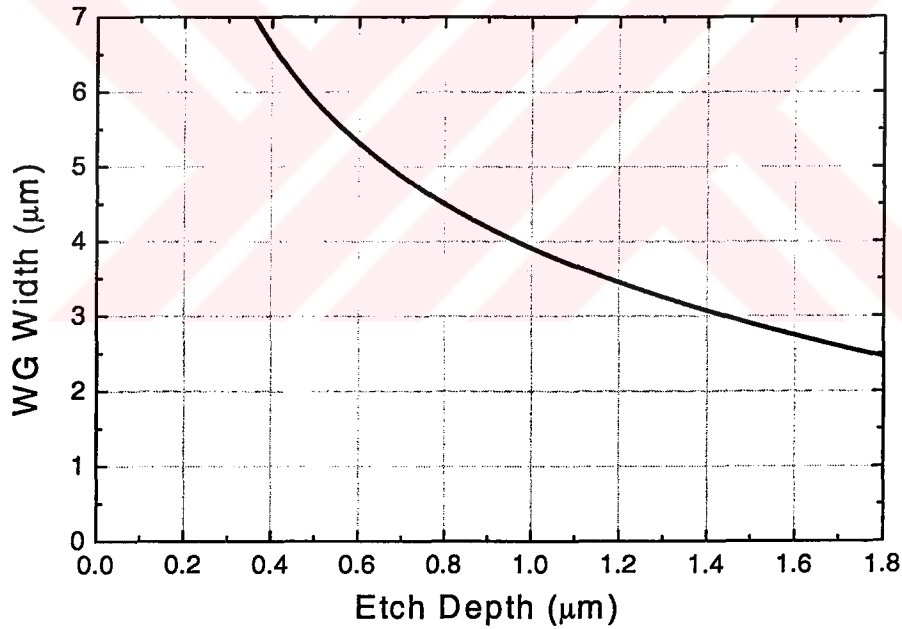


Figure 4. 5: Single mode condition for SiON waveguide.

is single-mode one. It is evident that according to the theory, the waveguide width is more critical parameter than the etch depth (rib height). Based on these calculations, a structure for SiON ridge waveguide was determined with

the following parameters

$$\text{bottom cladding thickness } t_{bc} = 7.2\mu\text{m}$$

$$\text{upper cladding thickness } t_{uc} = 5.0\mu\text{m}$$

$$\text{core layer thickness } h_{core} = 0.9\mu\text{m}$$

$$\text{rib height } h_{rib} = 1.1\mu\text{m}$$

$$\text{rib width } \omega_{rib} = 3.0\mu\text{m}$$

The mode spectrum and shape of the waveguide were also verified occasionally by using a commercial BPM simulator. It employs numerical methods known as Beam Propagation Methods (BPM) which solve the scalar or vector wave equation for any structure defined by $n = n(x, y, z)$. BPM assumes small index differences along propagation direction z , and provided that the calculation grid sizes are sufficiently small it gives reasonable results. The SiON based waveguide with the above parameters was simulated by this method as well, yielding a single mode operation, in accordance with the effective index method. The mode shape of the TE fundamental mode as obtained by BPM is depicted in Figure 4.6.

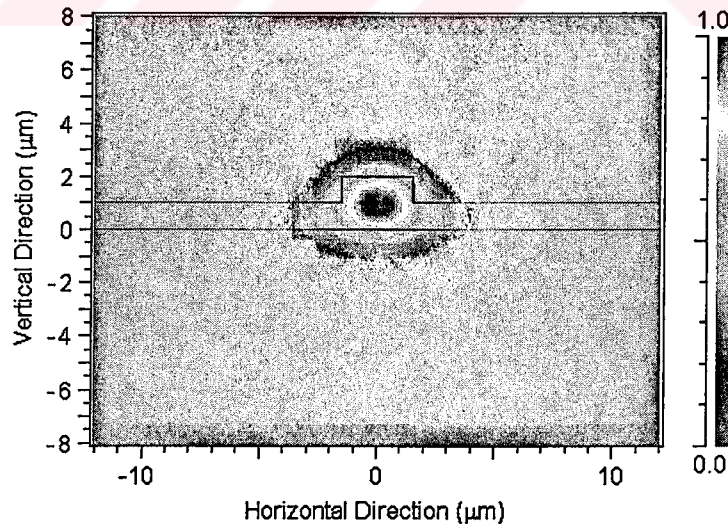


Figure 4. 6: The TE fundamental mode shape for SiON ridge waveguide as calculated by BPM.

4.1.3 Optical Loss Mechanisms

Together with the modal properties, the most important characteristic of a waveguide is its attenuation or loss, that a guided mode experiences as it travels through the waveguide. The loss arises mainly due to intrinsic material properties or imperfections that may come about during fabrication. A common measure of the *optical loss* is in dB/cm and it is defined as the logarithm of the power (intensity) ratio as

$$Loss (dB/cm) = \frac{10 \log \frac{P_{in}}{P_{out}}}{l} \quad (4. 17)$$

where l , P_{out} , and P_{in} are the waveguide length, output and input power, respectively. The measured total loss is the sum of the individual losses due to different mechanisms and below the ones responsible for the power attenuation in silicon oxynitride waveguides are described briefly.

Scattering Loss

A mode traveling in the guide experiences a scattering loss, in case of facing imperfections with index fluctuations of the order of the wavelength of light. According to the location of occurrence, the scattering losses in a waveguide can be divided into two as volume and surface scattering. In general, volume scattering results from imperfections such as voids or particle contaminations, that for our structure, may occur during the PECVD growth process. The loss due to them is proportional to the total number of scattering centers per unit length and is negligible compared to surface scattering loss.

The surface scattering loss might be of much more importance since the propagating wave interacts strongly with the surfaces that are more imperfect in terms of roughness. Namely, as the ridge waveguide is defined by etching, the surface may experience damage, in addition to which the side walls of the rib itself can be rather rough. In order to avoid these complications, a careful optimization of the reactive ion etching (RIE) process, applied for our waveguides, have to be worked out.

Although the both types of scattering occur at different locations, they result

from the same physical process of scattering of light. The power radiated as a result of this mechanism is given by the well-known expression of Rayleigh scattering

$$P_{rad} = \frac{\omega^4 (\Delta\epsilon)^2 E^2}{12\pi\epsilon_0 c^3} \quad (4.18)$$

where $\Delta\epsilon$ is the excess polarizability and E is the electric field component of light. The important feature of the equation is that the radiated power is inversely proportional to the fourth power of the wavelength.

Radiation Losses

Another type of energy loss in waveguides is in the form of loss of optical power from the mode into the media surrounding the waveguide. It can be observed in both slab and ridge waveguides in terms of leakage of the mode into the substrate or air. In fact, scattering losses may be included in this category, too. Radiation losses can also take place in the guides having sharp bend or curvature, but this is not an issue for our case. The SiO_2 lower cladding thickness of $7.2 \mu m$ ensures no radiation leakage in the our structure.

Losses Due to Vibrational Absorption

The main part of this study was devoted to the material treatment towards minimization of the optical losses at $\lambda = 1.55 \mu m$, caused by the first vibrational overtone of the N-H bond stretching vibration in the silicon oxynitride films. The detailed mechanism and characterization of this absorption has been studied in Chapter 3.

Having briefly discussed the loss types and mechanisms for silicon oxynitride based waveguides, we leave their measurement results to the next chapter.

4.2 Process Steps in Fabrication

The fabrication process of silicon oxynitride based waveguides was performed in class 100 and class 10000 clean room environment. The process consisted of one photolithography step, in which a light field mask containing straight waveguide strips varying from $2\ \mu\text{m}$ to $5\ \mu\text{m}$ was used. The whole process proved to be sensitive to the cleanliness of the environment and samples, which required special treatment at each step. Prior to processing, at each level samples were treated chemically as explained in section 2.1.4. The fabrication steps are depicted in Figure 4.7 and are explained below.

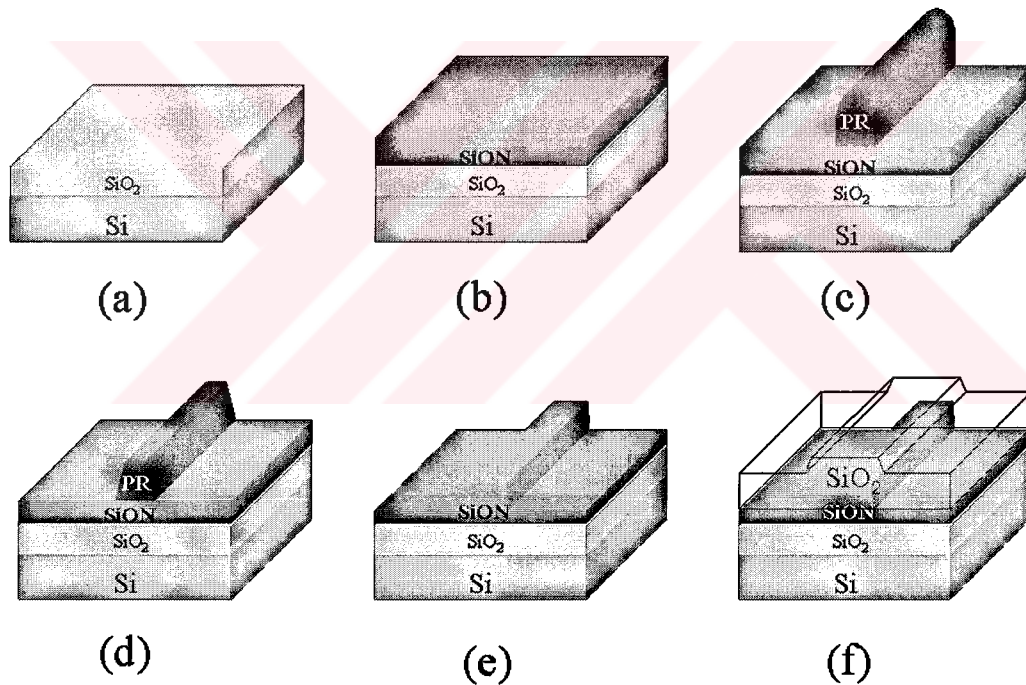


Figure 4. 7: Steps in the process of fabrication of SiON waveguides: (a) thermally oxidized SiO₂ and Si substrate, (b) PECVD growth of the SiON layer, (c) development of the coated photoresist, (d) structure after reactive ion etching, (e) removal of the photoresist mask and other organics, (f) PECVD of the upper cladding SiO_x layer.

PECVD Growth

Silicon dioxide layer of thickness $7.19 \pm 0.01 \mu\text{m}$ and index of refraction of 1.457 ± 0.001 , grown thermally on silicon (100) substrate (Fig.4.7 (a)) was used as an initial structure throughout the study. The silicon oxynitride layers of desired thickness ranging from $0.45 \mu\text{m}$ to $2.2 \mu\text{m}$ were grown by plasma enhanced chemical vapor deposition technique onto this structure (Fig.4.7 (b)). The chosen index difference between the lower cladding (SiO_2) and the SiON core layer was 0.04-0.05 and the optimum flow rates of the precursor gases N_2O and NH_3 , as determined by an extensive FTIR study, were 225 sccm and 15 sccm, respectively. The other parameters of the deposition process were discussed in section 2.4.

PECVD was also employed in the final step of the fabrication of SiON ridge waveguides in order to grow the upper cladding SiO_x layer (Fig. 4.7(f)). It was deposited with an index of refraction of 1.46 ± 0.01 and $5 \mu\text{m}$ thickness, with corresponding N_2O flow rate of 120 sccm at 350°C .

Waveguide Strip Definition

In order to transfer the waveguide strip pattern of the mask onto grown SiON layer, a standard photolithographic process is used. The cleaned sample is coated first with HMDS to improve the adhesion of the photoresist onto the surface of the film, spinning it at 4000 rpm for 40 seconds. The whole sample is then coated with AZ5214 photoresist (PR) material to serve as the masking layer, which ensures a thickness of $\sim 1.5\text{-}1.6 \mu\text{m}$, by again spinning at the same rate. Following the application of PR the sample is pre-baked for 50 sec. to harden the photoresist and remove any residual solvents. The sample is then placed for alignment of the mask on a Karl Suss MJS33 mask aligner, that is used for UV exposure at 365 nm Hg lamp. The exposure is done for 40 sec. at 4.0 mW optical power. The next step is to develop the exposed photoresist, which was done using 1:4 AZ400K : H_2O mixture for a duration of 50-60 sec, depending on the exposure time. Finally, the obtained structure is hard-baked at a hot plate at 120°C for two minutes (Fig.4.7 (c)).

The next step in the fabrication is to etch the SiON layer and define the strip of the ridge waveguide. For this purpose a reactive ion etching (RIE) system reactor has been used, by which SiON surface was etched using CHF_3 and O_2 gases.

In the process of dry plasma etching an rf glow discharge is excited in a high vacuum chamber between two electrodes, resulting in chemically reactive species. These species react with the SiON layer and form a volatile product, which is pumped out from the chamber. In brief, the basic steps of the process are: 1) generation of reactive species from CHF_3 and O_2 by the plasma; 2) transportation of these species to the surface of SiON layer; 3) adsorption of them on the surface; 4) formation of a volatile product as a result of chemical reaction; 5) desorption of the by-products from the surface and pump-out. The overall process is a competition between physical sputtering and plasma etching which favor anisotropic and isotropic etch, respectively.

The RIE process may have some side effects such as damage of the film surface, fast etching (low selectivity) of the PR mask layer, rough sidewalls of the defined rib, or PR sputtering and deposition. A control over all these parameters should be established by careful optimization of the process conditions for a successful waveguide fabrication. A study towards achieving this goal was performed by manipulating the variable parameters as indicated in Table 4.1. The first characterization was done by varying the CHF_3 flow rate, while keeping

CHF_3 flow rate	30-250 sccm
O_2 flow rate	10 sccm
Process pressure	20-100 μbarr
RF power	50-300 W
Process time	20 min.

Table 4.1: Process parameters used in characterization of reactive ion etching of SiON layers.

the flow rate of O_2 constant at 10 sccm, RF power at 51 W, process pressure at 50 μbarr . The obtained results for the etch rate variations are plotted in Figure 4.8. Next characterized parameters were the process pressure, in which the CHF_3 , O_2

flow rates and RF power were kept at 150 sccm, 10 sccm, and 51 W, respectively, and the RF power. The results of the etch rate variations are depicted in Figures 4.9 and 4.10.

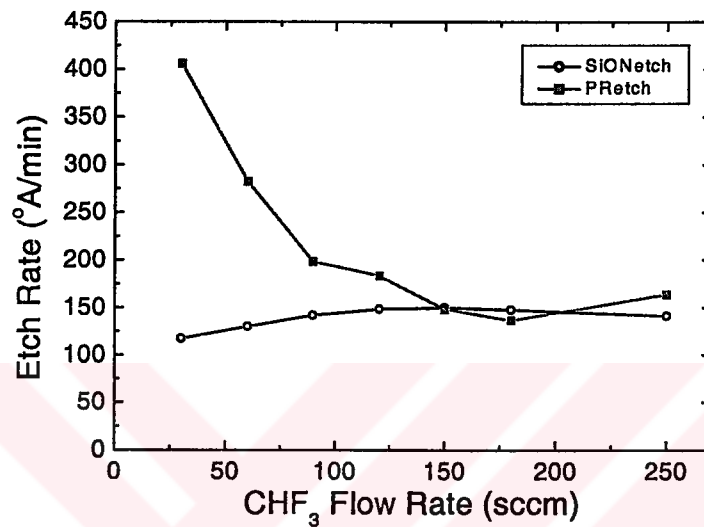


Figure 4. 8: SiON and photoresist mask etch rate variations with CHF₃ flow change.

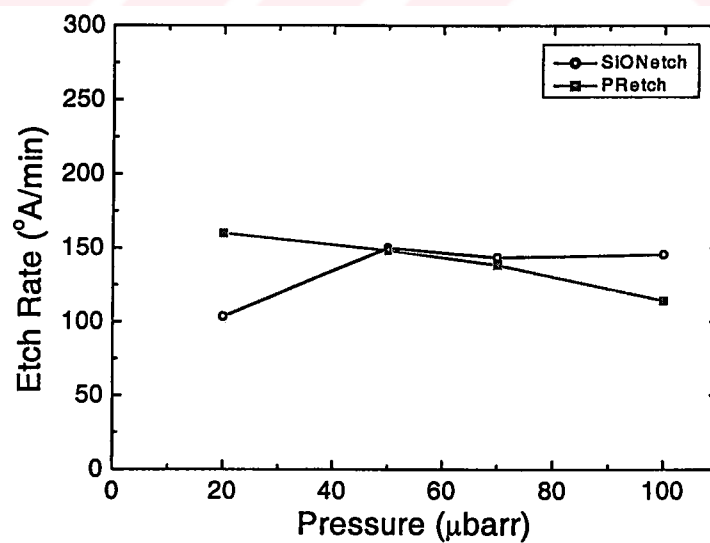


Figure 4. 9: SiON and PR etch rate variations with process pressure.

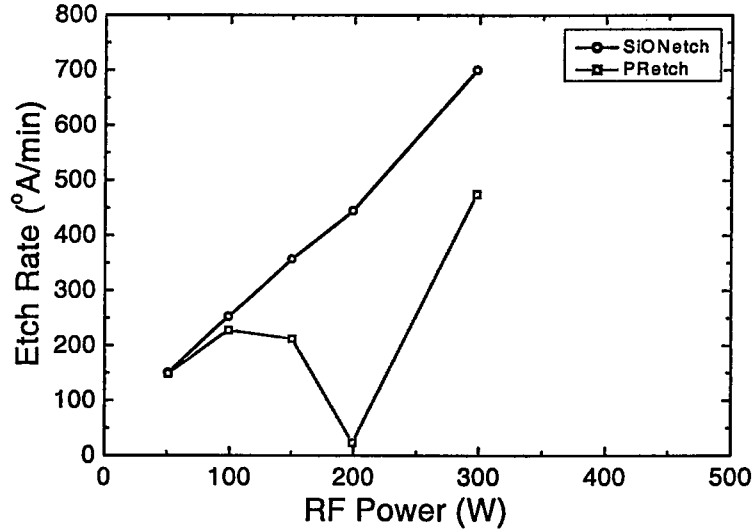


Figure 4. 10: SiON and PR etch rate variations with RF power.

Some of the etch rates were chosen to be the potential parameters for the strip definition process for *annealed SiON layers* and were analyzed by scanning electron microscopy (SEM). Interesting results were obtained, in which two of them worth mentioning. The first, at conditions of 100 W RF power, process pressure of 50 μ barr and CHF_3/O_2 flow ratio of 150/5, resulted in reactive chemical etch domination, creating rounded walls (see Figure 4.11). Next, in all the samples, although to different extent, there was observed a partial sputtering of the PR mask and possibly additional polymer deposition. An extreme example is given in Figure 4.12 where furthermore the side walls appear to be wriggled.

Finally, the parameters of CHF_3 and O_2 flow, RF power, and pressure of 150 sccm, 10 sccm, 99 W, and 50 μ barr were chosen to give the best results. Even in these conditions some leftover remained on the top of etched strip. However, they were removed by 20 min. O_2 plasma etch, after which the desired profile was obtained (see Figure 4.13). A delicate point is that production of waveguide strips having some degree of inclination was aimed. The driving reason behind this was to eliminate the problem of step coverage, during the final step of PECVD deposition of the upper cladding SiO_x layer. Therefore, we were successful in

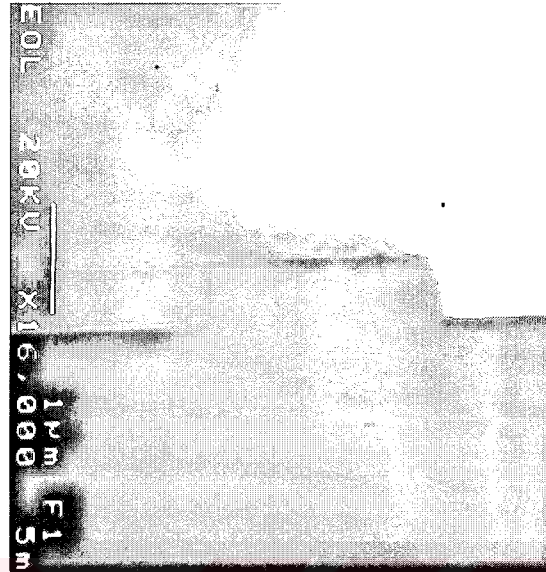


Figure 4. 11: SEM cross section of RIE etched rib with residual polymer on top .

fabricating strip side walls of $\sim 70^\circ$ inclination, with good uniformity.



Figure 4. 12: SEM picture of polymer on RIE etched SiON rib.



Figure 4. 13: Optimum RIE etch result.

Chapter 5

Waveguide Characterization and Measurements

Completing the fabrication of slab and ridge waveguides, that rely on silicon oxynitride technology, their characterizations are to be studied. In this chapter the techniques used in loss characterization of the mentioned structures are discussed. An attempt is made towards relating the absorption properties of the slab waveguides annealed at different temperatures with their corresponding N-H bond concentrations. Finally, the loss results of the ridge waveguides are discussed.

5.1 Optical Propagation Loss Characterization for SiON Slab Waveguides

Experimental Setup

The variation of the N-H bond concentration for a number of as deposited and annealed samples was monitored with the results presented in chapter 3. Now, any possible correlation of this concentration with the optical absorption in SiON waveguides should be checked. The best method for this is to measure the propagation losses for silicon oxynitride slab waveguides with the structure of

Figure 4.7 (b). Among the various methods of loss measurement a scanning detector system was used, which was capable of measuring the variation in the power of scattered light from the surface of the waveguide. In this setup any guided mode could be excited by making use of a prism coupler. A prism coupler has a structure as in Figure 5.1, where the prism is located at a distance h from the surface. A beam of light is directed into the prism with refractive index n_p

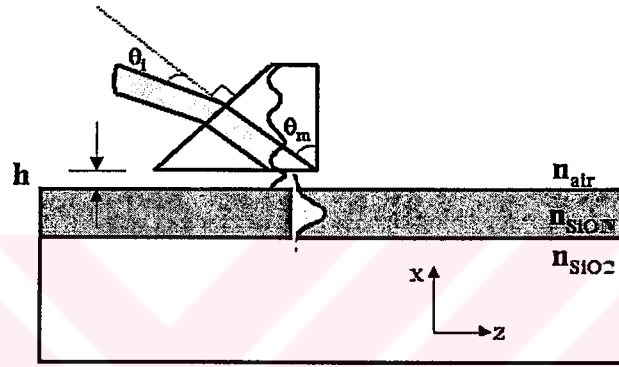


Figure 5. 1: Schematics of a prism coupler.

such that a total internal reflection occurs at the air-prism interface. The index of refraction of the film is n_{SiON} , that of the lower cladding SiO_2 layer n_{SiO_2} and for the outer region n_{air} was taken to be equal to one. The condition for total internal reflection is satisfied when ⁵³

$$\theta_p > \sin^{-1}\left(\frac{n_{air}}{n_p}\right) \quad (5. 1)$$

This gives rise to standing wave formation inside the prism, the k vector of which is given by

$$\begin{aligned} \vec{k}_0 n_p &= n_p(\pm k_x \hat{x} + k_z \hat{z}) \\ &= n_p(\pm k_0 \cos \theta_p \hat{x} + k_0 \sin \theta_p \hat{z}) \end{aligned} \quad (5. 2)$$

Outside the prism the field decays exponentially, but the z component of the wavevector remains the same. As evident from equation 5.2, k depends on the angle of incidence θ_p , which may enable us to adjust and possibly match the

velocity of propagation of the wave in the prism with that in the guide. The condition of matching of the β terms is given by

$$k_0 n_p \sin \theta_p = \beta_f \quad (5.3)$$

β_f being the propagation constant in the SiON film. Therefore, at an angle θ_p a coherent coupling of energy is achieved. By changing θ_p it is possible to select the desired mode of excitation. Few important points should be mentioned. First, the most effective coupling occurs when the spacing of the air gap h is on the order of one-fourth of the vacuum optical wavelength, and next, due to the above mentioned conditions the index of refraction of the prism should be greater than that of the film. In our measurements a LaSF crystal with $n_{TE} = n_{TM} = 1.875$ was used for excitation of the fundamental mode in the silicon oxynitride slab waveguide.

In the following measurements we have excited the TE_0 fundamental mode and made the all the characterizations on it. After the mode was forced to propagate along the guide an InGaAs photodetector was used to trace the power of the scattered light throughout the film. The whole experimental setup is diagrammed in Figure 5.2.

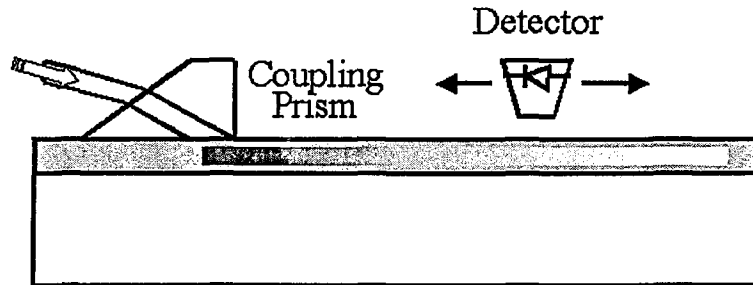


Figure 5. 2: The experimental setup used for propagation loss measurement.

Measurement Results

In order to trace the propagation loss, a study on five samples was performed. They had the following annealing conditions: sample 1 - as deposited; sample 2

- 800 °C for two hrs.; sample 3 - 900 °C for two hrs.; sample 4 - 1000 °C for two hrs.; sample 5 - 1150 °C for four hrs. The samples were deposited with identical parameters as the sample sion2 of section 3.6.3, i.e. N_2O and NH_3 flow of 225 sccm and 15 sccm, respectively. However, their sizes were approximately 30 mm $\times$ 60 mm, due to the restrictions of the loss measurement setup. The as deposited SiON layer thicknesses were $\sim 2.1 \mu\text{m}$, which supported two modes at $\lambda = 1.55 \mu\text{m}$. In the experiments a HeNe laser at $\lambda = 1.53 \mu\text{m}$ was used to couple light and excite the fundamental TE_0 mode in all the waveguides. A typical scattered light power profile along the guide is plotted in Figure 5.3 for the sample 3. The

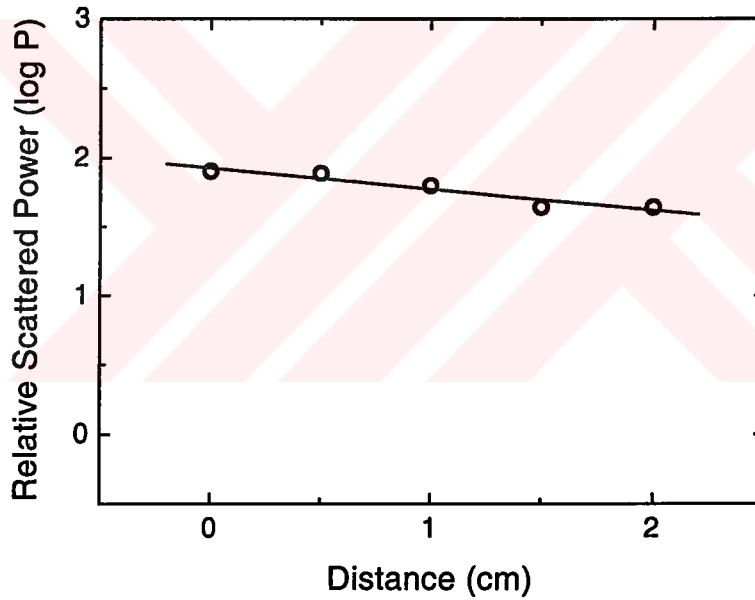


Figure 5. 3: Variation of the logarithm of the radiated power as a function of propagation length along the waveguide surface.

relative scattered power is plotted in in log P units, so as to the slope of the graph to give a value proportional to the propagation loss in dB/cm. The horizontal scale in the graph corresponds to the distance from arbitrary point close to the coupling prism. The propagation loss results, for the slab waveguides, together with their estimated N-H bond concentration are tabulated below.

Annealing Temperature °C	Propagation Loss dB/cm	[N-H] $\times 10^{22} \text{cm}^{-3}$
as deposited	-	0.52
800	3.7 ± 0.3	0.20
900	1.5 ± 0.3	0.09
1000	0.7 ± 0.3	-
1150 (4hrs)	0.01 ± 0.3	-

Table 5.1: Correlation between the N-H bond concentration and the loss profile of the SiON slab waveguides.

The accuracy of the measurements was estimated using the standard deviation of the detected scattered power during a scan on a specific sample. The loss result for the as-deposited sample could not be measured, which we account for the presence of large number of scattering centers in the film. Obviously, the quality of the SiON layers improves with annealing at higher temperatures, as was shown by the FTIR analysis and which is once more proved with the loss measurements.

Otherwise, we observe a steady trend of decrease in the propagation loss for the SiON layers with the corresponding decrease in their N-H bond concentration. Namely, the optical loss and N-H bond concentration ratio for the samples annealed at 800 °C and 900 °C are 2.5 and 2.2, respectively and are in a good agreement. Furthermore, an additional decrease in the loss is observed after an annealing at 1150 °C. This indicates a decrease in the N-H bond concentration, although there was none according to FTIR transmittance experiments, but whose existence was shown by ATR technique.

5.2 Ridge Waveguide Loss Characterization

Having optimized the propagation losses of silicon oxynitride slab waveguides, a fabrication and characterization of the aimed low loss ridge waveguides utilizing the same material technology follows. The process steps of their fabrication were discussed in section 4.2. Below, we deal with their insertion and propagation loss characteristics.

Experimental Setup

After fabrication of the single mode ridge waveguides and the optical cleavage of their ends, their loss characteristics were analyzed by making use of the so called *butt coupling* method. The distinguishing feature of this technique is that the light is coupled into the waveguide by a single mode fiber, an outcoupled by a lens which in turn directs the collected light to a Ge photodiode (see Figure 5.4). As a light source operating at $1.55\ \mu\text{m}$, a distributed feedback laser was used

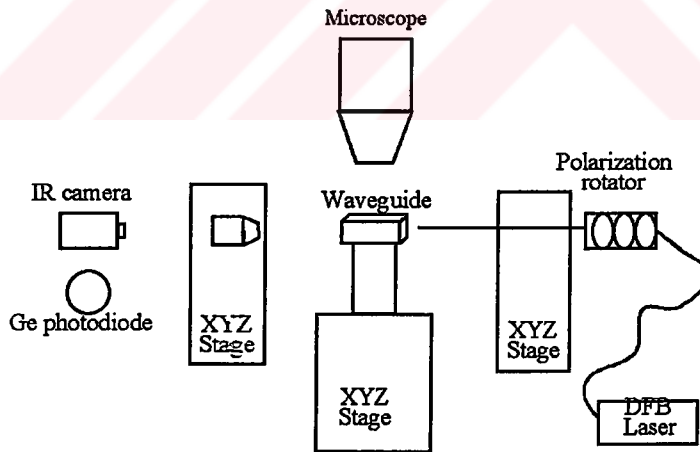


Figure 5. 4: Optical measurement setup .

under 27 mA current and $25\ ^\circ\text{C}$ temperature. It was attached to a fiber having polarization control module that allowed to select TE or TM polarized light. In addition, the sample was placed on an xyz translational stage enabling efficient coupling of the light into the guides. As for the parameters measured, we could

monitor the optical power transmitted using the Ge photodetector or the mode profile by an IR camera working in coordination with a video capture PC card.

Characterization Results

The single mode condition for the SiON waveguides with ridge height of $1.05\ \mu\text{m}$ was verified, as had been simulated by BPM (see Fig. 4.6). Experimentally, the well known method of directing the laser light in all possible off-axis positions in order to excite any possible higher order modes was employed. As expected, all the attempts resulted only in single mode propagation profile. An example of the mode profile as captured via the IR camera is given in Figure 5.5.

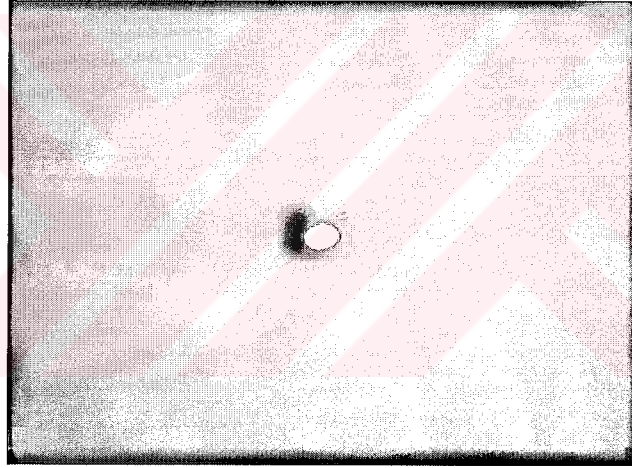


Figure 5. 5: View of TM_0 mode as captured by IR camera.

Following the single mode verification, an insertion loss measurement on the SiON ridge waveguides was performed. The insertion loss is measured in dB and includes the propagation loss, as well as the fiber to guide coupling loss. Applying the cut-back method, identical waveguides at different lengths were characterized. The measured values for waveguides of length of 12 mm, 8.5 mm, and 3.5 mm are given in Table 5.2 and are plotted in Figure 5.6.

Applying a linear fit to the loss data in the figure, from the slope we get the value of propagation loss of the ridge waveguides for TE and TM polarizations

	12 mm	8.5 mm	3.5 mm
TE	11.3 ± 0.6 dB	10.6 ± 0.5 dB	10.8 ± 0.4 dB
TM	11.3 ± 0.2 dB	9.9 ± 0.3 dB	10.7 ± 0.4 dB

Table 5.2: Insertion loss results for the TE and TM input polarized light for identical waveguides of different length.

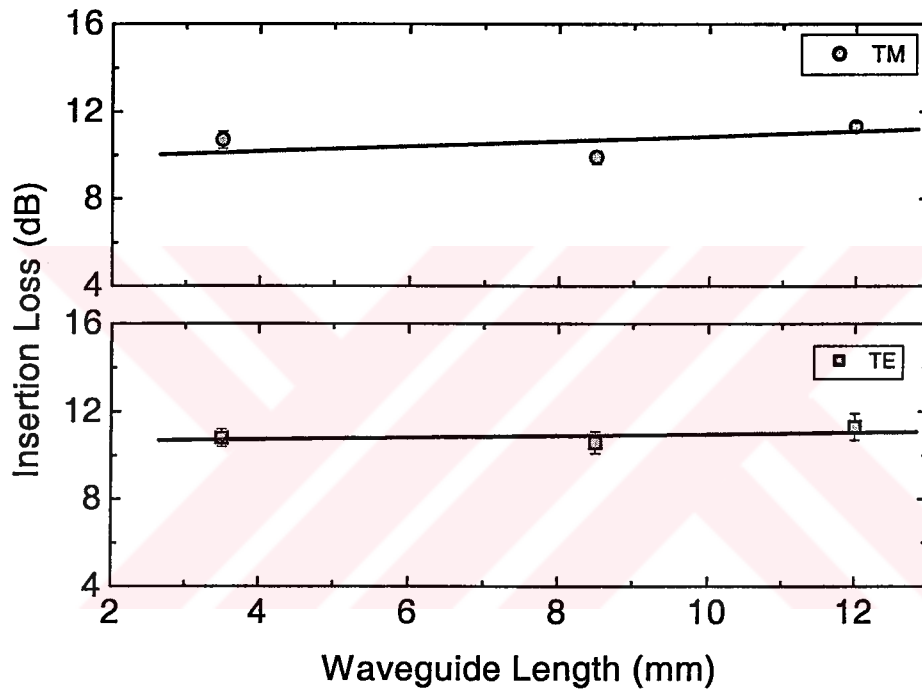


Figure 5. 6: Plot of insertion loss versus the waveguide length and estimation of the propagation loss by a linear fit.

as

$$TE : \text{Propagation Loss} = 0.46 \pm 0.33 \text{ dB/cm}$$

$$TM : \text{Propagation Loss} = 0.47 \pm 0.39 \text{ dB/cm}$$

Analysis of the results leads to a conclusion that the fiber coupling loss is about 4.9 dB/facet, which points out to the mode profile mismatch and reflection losses in the interface of the guide and fiber.

Comparing the propagation losses of the ridge waveguides with the slab guides

we see that the first ones' values seem to be at least of order of two larger. This difference is attributed mainly to two facts. First, after the growth of the upper cladding SiO_x layer the whole structure was not annealed. However, part of the mode profile is propagating in this layer, which contains certain amount of N-H bonds that are expected to absorb at $\lambda = 1.55 \mu\text{m}$. As an example, consider the TM_0 mode whose mode profile is larger than that of TE_0 . Since it propagates with a larger cross section into the upper SiO_x layer its loss is expected and is slightly larger than that of the TE mode. The other apparent cause for the greater loss in the ridge waveguides comes again from the upper oxide layer. As it is clearly seen from Figure 5.7, two small slit regions appear in the oxide layer,

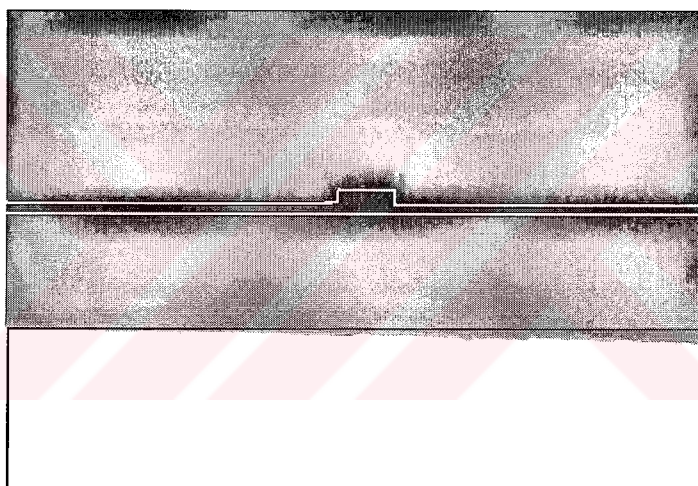


Figure 5. 7: Optical microscope image of the "slit" formation during the growth of the upper SiO_x cladding layer (approximate position of the SiON layer is outlined).

beginning from the upper corners of the ridge of the guide. This nonuniformity is expected to act as scattering center, thus contributing to the propagation loss increase.

Finally, evaluating the measured loss results, good values had been achieved for all loss types, namely insertion, and slab and ridge waveguide propagation losses. The reported best values^{20,29} for the propagation loss in slab waveguides are $1.1 \text{ dB/cm} \pm 0.2$ and $0.15 \pm 0.05 \text{ dB/cm}$, and for the ridge waveguides the

corresponding loss value is 0.2 dB/cm. This gives us direction for future study, i.e. to eliminate the excess absorption losses due to the SiO_x upper cladding layer.



Chapter 6

Conclusions and Future Prospects

In accordance with the main goal of this work, i.e. utilization of the silicon oxynitride technology and its optimization for applications in optical waveguides, an emphasis was given to the structural and optical properties of silicon oxynitride layers.

In the growth of silicon oxynitride films a standard PECVD technique was employed. For better understanding of the growth conditions and its compositional properties, attention was also given to silicon oxide and nitride films along with oxynitride layers. The silicon oxynitride films were deposited by varying the flow rates of N_2O and NH_3 precursor gases and keeping that of 2% SiH_4/N_2 constant at 180 sccm. The refractive index of the layers could be varied between 2.0 and 1.47 by changing the flow rates of the precursor gases.

The structural properties of these three types of films were investigated via Fourier transform infrared transmission spectroscopy. A special attention was given to the absorption band of N-H bond stretching vibration, since its first overtone is known to be the main cause of the optical absorption at $1.55 \mu\text{m}$ wavelength. For silicon oxide films its concentration was found to vary between $0.74 \times 10^{22} \text{ cm}^{-3}$ and $0.04 \times 10^{22} \text{ cm}^{-3}$, while this bond variation for silicon nitride layers was between $9.59 \times 10^{22} \text{ cm}^{-3}$ and $7.94 \times 10^{22} \text{ cm}^{-3}$. For the investigated silicon oxynitride films the corresponding variation of the N-H bond was observed to be $0.63\text{-}1.66 \times 10^{22} \text{ cm}^{-3}$.

Using the obtained results of refractive index and compositional characterization, a silicon oxynitride film with $n=1.50$ was chosen as a material to be used for core layer in single mode optical waveguide fabrication. The next concern was to reduce or eliminate the N-H related bond from the structure of this material. For this reason, an annealing study was performed using a furnace whose temperature profile could be controlled. Several annealing treatments were done at 800 °C, 900 °C, 1000 °C, and 1100 °C. As a result, we were able to reduce the N-H stretching bond concentration from $0.52 \times 10^{22} \text{ cm}^{-3}$ to a value $< 0.05 \times 10^{22} \text{ cm}^{-3}$, which is below the detection limits of the FTIR transmittance measurements.

In order to further verify the non-existence of this bonding type in the structure, an attenuated total reflectance (ATR) Fourier transform infrared technique was employed. This method has an advantage that it penetrates to only $\sim 2\mu\text{m}$ into the film from the Si/SiON interface, and thus might be capable of detecting any residual hydrogen bonding present at the interface, due to multiple reflections. Actually this was the case since there was still some amount of N-H stretching bonds observed even after the annealing temperature of 1000 °C. Moreover, another interesting feature was that some Si-H related bonding was detected at the interface by this method.

Furthermore, applying the same annealing treatment series as for the FTIR characterization, the correlation between the N-H bond concentration and the absorption loss was demonstrated for planar waveguides. By using prism coupling method and tracing the scattered light with a photodetector low loss slab waveguides were demonstrated. Finally, a single mode ridge waveguide was fabricated by optimizing the reactive ion etching process. Its insertion loss was measured to be $\sim 10 \text{ dB}$, and the propagation loss was at the order of 0.5 dB/cm . The non-competite step coverage, and inclusion of relatively large number of N-H bonds in the upper cladding SiO_x layer, together with the wall roughness of the etched rib were assigned as the source of the existing difference between the slab and ridge waveguides.

This has pointed out us a direction towards future study, in which further annealing treatment of the waveguide structure may be optimized in order to

decrease the propagation losses further. The silicon oxynitride technology is promising in many applications such as fabrication of multimode interference devices (MMI), Mach-Zender interferometers, etc, which constitute the potential fields of study in near future.



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