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**NANYANG
TECHNOLOGICAL
UNIVERSITY**

SINGAPORE

**A Novel Germanium-on-Silicon Nitride Platform
for Mid-IR Sensing**

LI WEI

SCHOOL OF ELECTRICAL AND ELECTRONIC ENGINEERING

**A thesis submitted to the Nanyang Technological University in partial
fulfillment of the requirement for the degree of Doctor of Philosophy**

2018

Statement of Originality

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research, is free of plagiarised materials, and has not been submitted for a higher degree to any other University or Institution.

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Supervisor Declaration Statement

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I have reviewed the content and presentation style of this thesis and declare it is free of plagiarism and of sufficient grammatical clarity to be examined. To the best of my knowledge, the research and writing are those of the candidate except as acknowledged in the Author Attribution Statement. I confirm that the investigations were conducted in accord with the ethics policies and integrity standards of Nanyang Technological University and that the research data are presented honestly and without prejudice.

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Tan Chuan Seng

Authorship Attribution Statement

This thesis contains material from 6 papers published in the following peer-reviewed journals / from papers accepted at conferences in which I am listed as an author.

Chapter 3 and Chapter 4 are published as *Li, Wei, P. Anantha, Shuyu Bao, Kwang Hong Lee, Xin Guo, Ting Hu, Lin Zhang, Hong Wang, Richard Soref, and Chuan Seng Tan. "Germanium-on-silicon nitride waveguides for mid-infrared integrated photonics." Applied Physics Letters 109, no. 24 (2016): 241101.*

- A/Prof Tan Chuan Seng provided the initial project direction and edited the manuscript drafts.
- I prepared the manuscript drafts.
- Dr Anantha P revised the manuscript.
- I conducted the fabrication of the Ge-on-SiN platform through wafer bonding and layer transfer technology. Single-mode strip waveguide and ultra-compact bent waveguide were designed and simulated using Lumerical FDTD. After the modelling, the strip waveguide and bent waveguide were fabricated in clean room and characterized by me.
- Dr Bao Shuyu and Dr Lee Kwang Hong helped to obtain the SEM images of the fabricated waveguides on GON platform and gave suggestions during the GON wafer fabrication process.
- Guo Xin built the measurement setup for characterization of passive devices so that the propagation loss and bending loss of the waveguides on GON platform can be measured.
- Dr Hu Ting gave constructive advice for the modelling and simulation.
- Dr Zhang Lin helped with the waveguide fabrication.
- A/Prof Wang Hong was kind to let me use their measurement setup.

- Prof Richard Soref provided the theoretical direction for using GON for Mid-infrared photonics.

Li, Wei, P. Anantha, Shuyu Bao, Kwang Hong Lee, Xin Guo, Hong Wang, Richard Soref, and Chuan Seng Tan. "Modeling and fabrication of Ge-on-Si $3N/4$ for low bend-loss waveguides." In 2016 IEEE 13th International Conference on Group IV Photonics (GFP), pp. 32-33. IEEE, 2016.

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- Dr Qiu Haodong conducted the E-beam lithography process.
- Dr Zhang Lin helped with the fabrication process.
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- Dr Anantha P prepared the manuscript drafts and conducted the fabrication process of the rib waveguide on Ge-on-Si platform.
- Dr Zhang Lin helped with the fabrication process of the waveguides.
- I conducted the characterization of the fabricated waveguides.

- Dr Qiu Haodong conducted the E-beam lithography process.
- Dr Chong Gang Yih conducted the Deep-RIE process.
- Dr Callum G Littlejohns helped with the waveguide fabrication process.
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Chapter 5 is published as *Li, Wei, P. Anantha, Kwang Hong Lee, Hao Dong Qiu, Xin Guo, Simon Chun Kiat Goh, Lin Zhang, Hong Wang, Richard A. Soref, and Chuan Seng Tan. "Spiral waveguides on germanium-on-silicon nitride platform for mid-IR sensing applications." IEEE Photonics Journal 10, no. 3 (2018): 1-7.*

- A/Prof Tan Chuan Seng provided the initial project direction and edited the manuscript drafts.
- I prepared the manuscript drafts. The manuscript was revised by Dr Anantha P.
- I conducted the fabrication of the spiral waveguide sensor on Ge-on-SiN. Single-mode spiral waveguide sensor was designed and simulated using Lumerical FDTD. After the modelling, the spiral waveguide sensor was fabrication using the standard CMOS process. Fluidic chamber made of PDMS was fabricated and integrated with the sensor chip and the IPA liquid sensing experiments were conducted by me.
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- Guo Xin built the measurement setup for characterization of passive devices so that the propagation loss and bending loss of the waveguides on GON platform can be measured.
- Dr Zhang Lin helped with the waveguide fabrication.
- Dr Qiu Haodong conducted the E-beam lithography process.
- Simon Chun Kiat Goh conducted FTIR on the IPA liquid to verify it has an absorption peak within the wavelength that we used.
- A/Prof Wang Hong was kind to let me use their measurement setup.
- Prof Richard Soref provided the theoretical direction for using GON for Mid-infrared photonics.

Li, Wei, P. Anantha, Kwang Hong Lee, Jin Zhou, Xin Guo, Hong Wang, and Chuan Seng Tan. "Germanium-on-insulator Pedestal Waveguide for Midinfrared Sensing Applications." In Signal Processing in Photonic Communications, pp. JTU2A-58. Optical Society of America, 2018.

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- Guo Xin built the measurement setup for characterization of passive devices so that the propagation loss and bending loss of the waveguides on GON platform can be measured.
- Zhou Jin conducted the E-beam lithography process after Dr Qiu Haodong left NTU.
- A/Prof Wang Hong was kind to let me use their measurement setup.

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Abstract

In recent years, the wavelength range over which silicon photonics can operate has been extended to the mid-infrared (IR) regions (2 to 20 μm). Since many molecules and substances have their specific absorption peaks in the Mid-IR range, this has provided us with a good opportunity to use silicon photonic device as a sensor to trace elements of chemicals in either gas, liquid or solid phases. In addition, Mid-IR also has many potential applications in free space data communication, IR imaging of biological tissues, spectroscopy and many others.

Conventional Germanium (Ge) on Silicon (Si) structure (GOS) has a small core/clad refractive index contrast which makes a compact design of sensing devices difficult to realize. The silicon on insulator (SOI) structure is a very mature and well established platform. However, SiO_2 is only transparent up to about 3.8 μm . When Ge on SiO_2 structure is used for sensing, absorption by the SiO_2 layer degrades the performance of such devices. Therefore, the main objective of this doctoral work is to realize a scalable and CMOS compatible germanium-on-insulator platform with wide transparency range in the Mid-IR as well as a large core-clad refractive index contrast in order to realize high performance and compact devices at low cost. Silicon Nitride (SiN_x) is known to be transparent up to 9 μm and it can provide a significant refractive index contrast with Ge. Therefore, Ge-on- SiN_x (GON) is proposed as a possible structure for sensing application in the Mid-IR range. However, high-quality single crystal Ge cannot be directly deposited on buried SiN_x layer as SiN_x is an amorphous material. In this thesis, a novel method enabled by wafer bonding and layer transfer is used to obtain a high quality Ge on SiN_x structure. The fabrication processes are discussed and optimized to obtain a Ge layer with low defect level (threading dislocation density $\sim 6 \times 10^6 \text{ cm}^{-2}$) and an inherent tensile strain of 0.13%. In order to characterize the passive devices fabricated on GON, an optical testing platform has been built up exclusively for Mid-IR applications ranging from 3.65 to 3.90 μm . After the preparation of the engineered wafer and the built-up of testing platform, systematic studies consist of modeling by finite difference time domain (FDTD) method, fabrication and characterization of the

strip waveguides and bent waveguides on GON and on GOS wafer, respectively, have been carried out. The simulation results show that the performance gap becomes larger as the radius of the bent waveguide decreases. In addition, the experimental results demonstrate that the propagation loss of the strip waveguide on GON is 3.35dB/cm at the wavelength of $3.80\ \mu\text{m}$ and the bending loss of the bent waveguide is 0.14dB/bend at the radius of $5\ \mu\text{m}$ while the bending loss of the bent waveguide on GOS is 2.54dB/bend at the same radius, which proves the proposal that GON platform can provide us with a more compact design of devices.

Mid-IR sensing applications have been explored using this novel GON platform as optical chemical detection is a fast growing market in many fields of use. The principle of the Mid-IR waveguide sensor is evanescent field sensing which is based on the interaction of the evanescent field of a waveguide mode with the surrounding substances and the resulting perturbation of the intensity of the mode at the output end of the waveguide. In order to improve the sensitivity, two strategies have been applied. One is to increase the length of waveguides thus to increase the interaction area with the surrounding chemicals. Another is to extend the evanescent field to enhance the absorption. Thus different structures of waveguides have been studied including spiral, pedestal, and slot waveguide either to increase the length or to extend the evanescent field. Spiral waveguide sensor on GON demonstrates a sensitivity of 5% to isopropanol in acetone solution compared to the counter-part on GOS whose sensitivity is 16% to the same solution. Pedestal waveguide sensor is capable of detecting 0.1% acetic acid mixed with water by undercutting the buried oxide layer and applying fundamental TM mode. This thesis provides deep insights, with experimental results, into the design and optimization of the Mid-IR waveguide sensors primarily on germanium-on-silicon nitride platform.

List of Publications

1. Li, Wei, P. Anantha, Shuyu Bao, Kwang Hong Lee, Xin Guo, Ting Hu, Lin Zhang, Hong Wang, Richard Soref, and Chuan Seng Tan. "Germanium-on-silicon nitride waveguides for mid-infrared integrated photonics." *Applied Physics Letters* 109, no. 24 (2016): 241101.
2. Lee, Kwang Hong, Shuyu Bao, Yiding Lin, Wei Li, P. Anantha, Lin Zhang, Yue Wang, Jurgen Michel, Eugene A. Fitzgerald, and Chuan Seng Tan. "Hetero-epitaxy of high quality germanium film on silicon substrate for optoelectronic integrated circuit applications." *Journal of Materials Research* 32, no. 21 (2017): 4025-4040.
3. Li, Wei, P. Anantha, Shuyu Bao, Kwang Hong Lee, Xin Guo, Hong Wang, Richard Soref, and Chuan Seng Tan. "Modeling and fabrication of Ge-on-Si N 4 for low bend-loss waveguides." In *Group IV Photonics (GFP), 2016 IEEE 13th International Conference on*, pp. 32-33. IEEE, 2016.
4. Li, Wei, P. Anantha, Kwang Hong Lee, Jin Zhou, Xin Guo, Hong Wang, and Chuan Seng Tan. "Germanium-on-insulator Pedestal Waveguide for Midinfrared Sensing Applications." In *Signal Processing in Photonic Communications*, pp. JTU2A-58. Optical Society of America, 2018.
5. Li, Wei, P. Anantha, Kwang Hong Lee, Hao Dong Qiu, Xin Guo, Simon Chun Kiat Goh, Lin Zhang, Hong Wang, Richard A. Soref, and Chuan Seng Tan. "Spiral Waveguides on Germanium-on-Silicon Nitride Platform for Mid-IR Sensing Applications." *IEEE Photonics Journal* 10, no. 3 (2018): 1-7.
6. Anantha, P., Lin Zhang, Wei Li, Xin Guo, Haodong Qiu, Gang Yih Chong, Callum G. Littlejohns et al. "Low propagation loss Ge-on-Si waveguides and their dependency on processing methods." In *Lasers and Electro-Optics Pacific Rim (CLEO-PR), 2017 Conference on*, pp. 1-3. IEEE, 2017.
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platform for mid-IR sensing applications." In *Photonics Conference (IPC), 2017 IEEE*, pp. 107-108. IEEE, 2017.

8. Son, BongKwon, Yiding Lin, Wei Li, Kwang Hong Lee, Lin Zhang, Tina Xin Guo, Hong Wang, Chuan Seng Tan. "Metal-semiconductor-metal germanium-tin photodetector on a GeSn-on-insulator platform." In *Photonics West, 2018 SPIE*. (Accepted)
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Chapter 1: Introduction

1.1 Motivations and Objectives of the Thesis

With the rapid development of technology, the critical size of transistors has already reached the nanometer range, and the number of transistors on chips is unbelievably tremendous currently [1]. Metal wires play a very important role in chips because they can connect different devices together in a single chip, or interconnecting between different chips. However, due to the rapidly-increasing number of transistors within a single chip, as well as the complexity of designs, the total length of metal wire is bound to become larger and larger. As a result, the total resistance increases. Hence, power consumption becomes a very critical challenge in today's semiconductor technology. Additionally, the capability of carrying information of electrons is not as good, compared with photons, and so people have tried to use optical interconnections in place of metal wires to connect different devices and chips, which can increase the volume of information transmitted in the chips and also lower power consumption [2]. The rapid development of photonic components also requires a high level of integration so that different photonic components can form a system to realize a certain function. This is why Silicon photonics, or Group IV photonics, has become such a hot research field in the last decades. Optical waveguides can replace metal wires to transmit signals with more volume, but also with less power consumption. In 2008, Shacham *et al.* predicted that the energy for transmitting signals can be decreased to 0.2 pJ/bit by using Si ring-resonator modulators and SiGe photodetectors [3]. Waveguides form another integration method between discrete photonic components in a single chip. In fact, the integration of discrete photonic components into a single chip has been a long-standing goal of integrated optics. The dominant platform that we use today for photonics is the silicon-on-insulator, otherwise known as SOI. The SOI platform is well-established, with all fabrication processes for silicon photonics being compatible with the CMOS process in foundries. Silicon photonics has already obtained many impressive achievements in high-performance arrayed waveguide gratings, resonators, modulators, and photo-detectors [4-7]. Recently, the first microprocessor integrated with silicon

photonics circuits was reported [8]. This single-chip microprocessor, which communicates directly using light at a wavelength of $1.55\ \mu\text{m}$, indicates a trend of the development of the semiconductor industry, as mentioned above. Silicon photonics on the SOI platform has a great performance at the wavelength range of 1.3 to $1.55\ \mu\text{m}$. However, when it comes to the mid-infrared range, the SOI platform seems a less proper choice, since silicon dioxide has its absorption peak in the mid-infrared range, resulting in a high transmission loss.

The mid-infrared (Mid-IR) range, defined here as 2 to $20\ \mu\text{m}$, has attracted much research interest because of its sensing applications and potentials in advanced telecommunications. Regarding sensing applications, this involves the detection of potentially lethal trace gases, which are the by-products of a standard process, harbingers of an unforeseen catastrophe, or representations of poor air quality in homes or commercial buildings. In addition, people are currently paying more attention to what they eat and drink, as health and food safety are becoming issues of concern with growing food safety problems, and a series of chronic diseases caused by excessive consumption, like diabetes. These challenges depicted in *Figure 1* require a fast and effective way of detection, which helps people respond to crises faster, as well as monitor food safety and how much nutrition they gain from food in real time.

Figure 1. Some challenges such as spill of toxic chemicals, air pollution, food safety, and excessive consumption of sugar that need to be addressed nowadays.

Fortunately, there are a number of relevant gases whose characteristic absorption lines lie in the Mid-IR wavelength range, as shown in *Figure 2* [9]. Besides gas sensing, many substances and molecules also have absorption peaks within this range, such as alcohol [10] and cocaine [11] (not shown in *Figure 2*). Thus, besides industry and commercial applications, mid-infrared sensing can play a very important role in biochemical, health, and environmental variables [12].

Figure 2. Chemical compounds and their characteristic absorption peaks in Mid-IR range [9].

However, many tools used for chemical detection are bulky, discrete, and expensive, failing to meet key requirements for a large number of applications. With the development of technology and society, devices aiming for detection and health monitoring have to be small, fast, wearable, cheap, and versatile. Conventional equipment, like the Fourier transform infrared spectroscopy, which needs experts to interpret the data, or discrete optical components that rely on assembly, do not meet these requirements. On the other hand, the full integration of all optical components, for sensing on the planar substrate at the chip level, would be a major advancement due to its higher performance, new functionalities, and higher reliability at much lower costs, smaller size and reduced weight and power consumption. The photonic integrated circuit is a feasible solution to realize all these requirements, as shown in *Figure 3*. By integrating the laser light source with a large tunable range, high-performance modulator, high-sensitivity waveguide sensor, photodetector, electronic drive circuit and read-out circuit, miniaturized sensing devices based on Group IV photonics for Mid-IR sensing can be realized.

Figure 3. 3D schematic of Photonic Integrated Circuit for Mid-IR sensing devices.

Currently, telecommunications through single mode fibers lie mainly in the near-infrared telecom wavelength region (1.3 to $1.55\ \mu\text{m}$). However, the maximum capacity of this wavelength range is not infinite; instead, it reaches a “capacity crunch” (see Figure 4). In order to meet the continued steep growth of transmitted data volumes across all media, a promising solution is to use new spectral bands, at more than $1.55\ \mu\text{m}$, for next-generation optical communication systems with ultra-low loss multi-mode photonic crystal optical fiber communications [13]. It is predicted that the capacity increase will accrue primarily in a new transmission wavelength window, centred at $2\ \mu\text{m}$.

Figure 4. Record data transmission capacity of fiber transmission system [13].

Since the mid-infrared range offers so many advantages, tremendous efforts have been put into the study of extending the operating wavelength of silicon photonics into this range. Silicon on insulation (SOI) has been the dominant platform in the semiconductor industry for many decades, and appears to be a good choice for mid-infrared photonic applications. The refractive index of Si is 3.4, whereas the refractive index of the under-cladding layer (SiO_2) is 1.4, hence the refractive index contrast between the core material and the cladding material is very large, allowing for a small radius of bent waveguides, as well as relatively small cross-sectional dimensions due to good confinement [14]. Thus, many photonic components with compact footprints can be realized on the SOI platform. However, the under-cladding layer—silicon dioxide—is only transparent up to about $3.8\ \mu\text{m}$, as shown in *Figure 5* [15, 16]. A light source with a wavelength larger than $3.8\ \mu\text{m}$ will be absorbed by the silicon dioxide layer, causing high loss during the light transmission. Therefore, the SOI platform is not suitable for longer wavelength operations. The Germanium-on-silicon platform is widely used currently, with Ge being transparent up to about $14\ \mu\text{m}$ and Si also being transparent to about $8\ \mu\text{m}$. Therefore, the Ge-on-Si platform is ideal for mid-infrared applications. However, the refractive indices of Ge and Si are 4.0 and 3.4 respectively. The refractive index contrast between the core and the cladding is only about 0.6, which is quite small compared to the SOI platform, which will make compact on-chip design difficult. Nowadays, the size of chip or device is an important consideration, with components preferred to be as small as possible.

Figure 5. Infrared wavelength range over which the materials are transparent. The white areas represent the transparent window of each material [16].

Therefore, this thesis aims at realizing a new platform that can operate over an acceptable wavelength range in Mid-IR, as well as has a large core-clad index contrast to realize compact footprints of devices and components, to make preparations for future photonic integrated circuits. Waveguides with different geometries for different applications are designed, modelled, and fabricated on the new platform. A testing optical table is built up for waveguide characterization at the Mid-IR wavelength. Lastly, Mid-IR sensing applications using the designed waveguide sensor are studied.

1.2 Scope and Contributions of the Thesis

In this doctoral work, a novel platform Germanium-on-Silicon nitride has been designed, fabricated and characterized by wafer bonding and layer transfer technique. Single-mode strip waveguide and ultra-compact bent waveguide with low bending loss has been designed, fabricated, and characterized. Mid-IR sensing application has been explored by realizing waveguide sensors on this novel platform.

In *Chapter 1*, the motivation and the objectives of this thesis are introduced.

In *Chapter 2*, a general overview of available platforms for Mid-IR photonics are presented. A brief introduction on the wafer bonding technique for realizing diversified platforms for Group IV photonics is made. Some achievements and results based on the waveguide sensor at Mid-IR wavelengths are also reviewed and concluded.

In *Chapter 3*, the fabrication process of the novel platform—Germanium-on-silicon nitride (GON)—is described, followed by the optimization process of how to improve bonding quality. The characterization results of the final bonded wafer are also presented.

In *Chapter 4*, the equipment and setup process of the optical table for waveguide loss measurement is introduced. The methods for propagation loss measurement are also described to prepare for the next waveguide characterization. The influence of the dry etching process on the etching profile and performance of the rib waveguide on the germanium-on-silicon platform was studied first, as the dry etching process is critical for low-loss waveguide fabrication. Then, the theoretical calculation, modelling, and

fabrication of the single-mode strip waveguide and ultra-low loss bent waveguides are presented.

In *Chapter 5*, Mid-IR sensing applications are explored, based on the novel germanium-on-silicon nitride as well as germanium-on-insulator platforms. Methods for improving the sensitivity of waveguide sensors, by either enlarging the total length of waveguides or extending the evanescent field fraction, are described. Chemicals like acetone, isopropanol, and acetic acid, that are normally used in foundries and food industries, were detected by our waveguide sensors on the novel platform at Mid-IR wavelengths.

In *Chapter 6*, conclusions are drawn and recommendations on future work are made.

The key contributions of this doctoral thesis are:

- A novel Germanium-on-Silicon nitride platform for Mid-IR photonics has been designed, fabricated and characterized.
- Ultra-compact bent waveguide with low bending loss for Mid-IR photonics has been designed, fabricated and characterized.
- Absorption based Mid-IR sensing application has been explored by applying waveguide sensor on the novel platform. The methods to further increase the sensitivity have also been studied.

Chapter 2: Overview of Si Photonics and Its Mid-IR Sensing Applications

2.1 Silicon Photonics

Silicon photonics has become a popular field of research in the last several years, especially its fundamental passive component—waveguides. Optical waveguides can be used in place of metal wires to transmit signals with more volume, but also with less power consumption. Waveguides also form a method of integration between discrete photonic components within a single chip. In fact, the monolithic integration of discrete photonic components into a single chip has been a long-standing goal of integrated optics. In previous silica-on-silicon technology, the waveguide was formed in a silica layer by doping it with phosphorus or germanium atoms [17]. Regarding this technology, an impressive level of integration can be achieved; for example, a 16x16 switch array on a single 6 inch silicon wafer [18]. However, further increases in integration density with this technology are restricted by the large minimal bending radius of silica waveguides, which is of the order of a few centimeters. A significant step toward much denser integration has been demonstrated with silicon oxynitride (SiON) technology [19]. A much higher index contrast is introduced between the core of the SiON waveguide and silica cladding, which allows the minimum bending radius to be reduced to below 1 mm. Further aggressive scaling is introduced by silicon-on-insulator (SOI) technology, where the waveguide is formed in a thin silicon layer. An extremely high refractive index contrast between the silicon core ($n=3.4$) and silica cladding layer ($n=1.4$) allows the waveguide core to be shrunk down to a submicron cross-section, while still maintaining single mode propagation at 1.3 to 1.5 micron telecommunications wavelengths. Such extreme light confinement allows the minimal bending radius to be reduced to the micron range. Currently, the most dominant platform in silicon photonics is the Silicon on Insulator (SOI). A buried oxide layer (BOX) with a typical thickness of 1 to 2 μm is sandwiched between the top guiding Si layer and the Si substrate. Different SOI thicknesses have been reported in the literature and used by foundries. Established silicon photonics foundries, such as IMEC and

IME, have converged to using SOI wafers with 220 nm-thick Si. Propagation losses as low as 0.6 dB/cm have been reported for 220 nm-thick SOI waveguides, at 1550 to 2000 nm. For shallow rib waveguides with 70 nm etch depth, the loss only ranges from 0.1 to 0.2 dB/cm [20]. Commercially-available optical transceiver cables from Luxtera or Molex are based on SOI, with 300 to 310 nm-thick Si, which was chosen to accommodate low-loss compact passive devices and for a bulk-like transistor process [21].

In 2006, Richard Soref proposed to extend the operation wavelength of this technology to the longer infrared region, as many attractive applications and markets lie in there, such as optical countermeasures, IR imaging of biological tissues, free-space data communication, and the most mature field of usage – the detection of trace elements of chemicals in either gaseous, liquid or solid phases. Among these applications, technical developments are mainly driven by optical chemical detection, which is a fast-growing market worth 7 billion US dollars, spanning many fields of use. Numerous research interests and efforts have been harnessed to find a suitable platform to extend the operation wavelength, as well as realize its full integration at the chip level. Instead of using discrete components for sensing, photonic integrated circuits (PICs) would be more effective, due to their high performance, new functionalities and higher reliability at lower costs, smaller size, as well as reduced weight and power consumption. Different materials have been used to form different core/clad stacks to extend the operation wavelength till Mid-IR, as well as provide a large core/clad refractive index contrast of the same. These platforms will be described in detail as follows.

2.1.1 Si-Based Platform

SOI is the most dominant and well-established platform for the CMOS industry and near-IR communication, owing to the availability of low-cost SOI wafers based on the SOITEC smart-cut process. The core material Si has a wide transparency range, of up to 7 μm , and can form a large refractive index contrast with the buried oxide layer. However, the buried oxide layer starts to absorb strongly beyond 3.8 μm . Roelkens *et al.* used the SOI for operations up to 3.8 μm , by increasing the thickness of both the Si

and oxide layers. This SOI buried oxide layer has a thickness of $3\text{ }\mu\text{m}$, with the Si layer having a thickness of 400 nm . Propagation losses of 3 dB/cm were reported at $3.8\text{ }\mu\text{m}$ for the waveguides [22]. Partially-etched rib waveguides on the SOI wafer, reported by Mashanovich *et al.*, had a propagation loss of $\sim 1.5\text{ dB/cm}$ at the wavelength of $3.8\text{ }\mu\text{m}$ and fully-etched strip waveguides had the lowest loss of 1.28 dB/cm [23]. An SOI vertical slot waveguide was proposed by Soler Penades *et al.* at $3.8\text{ }\mu\text{m}$. With a slot gap of around 80 nm etched in a 500-nm-thick Si layer, they achieved propagation losses as low as 1.4 dB/cm [24].

Many impressive results have been achieved using the SOI wafer; however, the natural optical property of the buried oxide layer limits the usages of the platform for even longer infrared regions beyond $3.8\text{ }\mu\text{m}$. Two main approaches have been investigated in order to extend the operation wavelength: the substitution of core or clad materials with larger transparency windows and the removal of the buried oxide layer to form air-suspended structures. In ref [25], a material stack silicon-on-silicon nitride (SON) was realized by the deposition of a thick SiN_x layer and its bonding to another Si die, to extend the operation wavelength by potentially up to $7\text{ }\mu\text{m}$. A rib waveguide was then fabricated on this SON structure with $2\text{ }\mu\text{m}$ rib thickness, $0.8\text{ }\mu\text{m}$ etch depth, and 2 to $2.5\text{ }\mu\text{m}$ rib width for single mode operations, as shown as *Figure 6* below. The propagation loss, of 5.2 dB/cm , was obtained at $3.39\text{ }\mu\text{m}$ wavelength.

Figure 6. Scanning Electron Microscope (SEM) image of a fabricated rib waveguide on SON platform [25].

Another research group chose sapphire instead of SiN_x to serve as the under-cladding layer. This material stack, called silicon-on-sapphire (SOS), has the advantage of offering the ability to build high-confinement, fully-etched waveguides from 1.1 to about $6.2\ \mu\text{m}$ [26]. Besides the advantage of enlarging the transparency window, SOS is particularly desirable for Mid-IR applications because of the lack of a high index substrate, which eliminates the issue of substrate leakage. The resistivity of the silicon used for the wafers was specified by the manufacturer to be $100\ \Omega\text{-cm}$, suggesting that optical loss due to free-carriers would be minimal. 600-nm-thick Si was deposited on the high-purity artificially grown sapphire crystals, and strip waveguides with a width of $1.8\ \mu\text{m}$ were fabricated and characterized. The resulting loss was 4.3 dB/cm at the wavelength of $4.5\ \mu\text{m}$. Li *et al.* have successfully reduced the propagation loss of the waveguide on SOS to 1.92 dB/cm at the wavelength of $5.18\ \mu\text{m}$, which has been the lowest value published [27].

Figure 7. A false-color scanning electron micrograph of the cleaved endfacet of a waveguide. Silicon is shown in green, and sapphire in blue [26].

To avoid SiO_2 absorption, a common approach is to suspend the Si structures by locally removing the buried oxide layer of the SOI wafer. There has been a report using a pedestal structure to reduce leakage to the Si substrate, by etching the Si isotropically to form a tip to support the strip planar waveguide above it [28]. The optical losses of this strip waveguide on pedestal Si support have been as low as 2.7 dB/cm at $3.7\ \mu\text{m}$, which is an adequate performance. However, the fabrication process of this structure is

quite complicated because of the isotropic etching of the Si. In order to form the pedestal support, the strip waveguide previously etched has to be coated with oxide to protect it from the subsequent isotropic etching. After the Si isotropic etching, the deposited oxide has to be removed by HF wet etching. These steps make such a structure impractical for high-quantity industry fabrications. Nevertheless, it does provide us with an idea that a pedestal structure realized on SOI, by simply wet etching the buried oxide, can in fact reduce the absorption of the oxide layer, thus extending the operation wavelength as well as reducing optical losses. This example of the SOI pedestal waveguide is illustrated in *Figure 8* below. In ref [29], Lin *et al.* partially removed the buried oxide layer, by using buffered oxide etching (BOE) to selectively etch the oxide after the strip Si waveguide patterning, to form the Si-on-pedestal SiO₂ structure. According to the simulation results presented in their paper, these waveguides saw a propagation loss which was 10 dB/cm lower, compared to other waveguides using planar SiO₂ cladding at the wavelength of 5 μm . However, the experimental data was not presented in their paper.

Figure 8. (a) Fabrication process of the SOI pedestal waveguide. (b) SEM image of the fabricated SOI pedestal waveguide with width of 3 μm , height of 2 μm , and notch width of 0.5 μm [29].

According to the modeling results, the best results were still obtained when the oxide was totally removed, but the question remains as how to realize a free-standing Si device layer when the buried oxide layer is totally removed. Shankar *et al.* suggested using photonic crystals, where the holes in the Si layer would allow the diluted HF

liquid to pass through and etch the SiO₂ beneath [25]. Motivated by this idea, Soler Penades *et al.* introduced a subwavelength lattice of holes along the sides of the wire waveguides, allowing the total removal of the BOX under the Si device layer while also ensuring mechanical stability. They termed this the subwavelength grating (SWG) waveguide. Subsequently, the removal of the buried oxide extended the operation wavelength by up to 8 μm . The thicknesses of the Si device layer and the BOX are 500 nm and 3 μm respectively; with the subwavelength lattice on the sides of the wire waveguide designed to act as a reflecting mirror to confine the guiding mode. The period of the SWG was determined by the formula: $\Lambda = l_{\text{Si}} + l_g < \Lambda_b = \lambda_0 / 2n_{\text{BF}}$, where l_{Si} , l_g are the Si strip and gap lengths in the subwavelength cladding respectively, and n_{BF} is the effective index of the fundamental Bloch-Floquet mode propagating in the waveguide. It had a period of 550 nm and diameter of 450 nm for the holes. This fabricated device is shown in *Figure 9*.

Figure 9. Top view SEM image of the SWG waveguide after immersion in 1:7 liquid HF for 30 min. In the top left inset a SEM image of the facet of a cleaved waveguide, an isotropic etching of the oxide can be observed from the points of entry of the etchant solution [30].

At the wavelength of 3.8 μm , the waveguide propagation loss measured 3.6 dB/cm. By improving the design and fabrication process, the same group reduced the propagation loss to 0.82 dB/cm at the same wavelength [31]. These researchers have also successfully extended the operation wavelength to an even longer region with this

suspended SWG waveguide, with the loss measuring 3.1 dB/cm at the wavelength of $7.67\ \mu\text{m}$ [30]. This was the first demonstration of low-loss Si waveguides at an extended wavelength. However, the main drawback of this technique is the need for high-resolution lithography equipment to pattern the thin (100 nm) Si grating arms.

A fully-suspended slot waveguide was realized on the SOI platform by applying a sub-wavelength grating to the sides of the slot waveguides. Slot waveguides are different from conventional wire or strip waveguides, as most of the light is guided in the low-index gap region of slot waveguides, which makes it suitable for sensing applications due to the large light-analyte interaction area. However, the losses of slot waveguides are very difficult to suppress, due to the same reason that most of the light is confined to the gap region, making it highly sensitive to imperfections in fabrication and environmental perturbations. Zhou *et al.* reported the propagation loss of the fully-suspended slot waveguides to be 2.8 dB/cm for a wavelength of $2.25\ \mu\text{m}$, which is impressive compared to previous results of 10 dB/cm [32, 33]. The scanning electron microscope (SEM) images of the fully-suspended slot waveguides are shown in *Figure 10*. The operation wavelength is expected to be further extended to a longer infrared region, and Mid-IR sensing applications can be explored using this technique.

Figure 10. (a) SEM top view of an asymmetric fully suspended slot waveguide. (b) Cross sectional view of a cleaved waveguide end [32].

The tremendous amount of work done on the development of Si-based platforms and devices has resulted in a library of high-performance components. However, the presence of SiO_2 in the substrate either constrains the operation range till about $3.8\ \mu\text{m}$,

or it imposes serious design limits and fabrication complexities for industries or foundries. In the domain of chemical sensing, these constraints substantially limit the suitability of these technologies in practical applications, which require longer wavelengths.

2.1.2 Ge and SiGe-Based Platform

Germanium (Ge) has a wide transparency window ranging from 2 to 15 μm , essentially covering the fingerprint region of most molecules. The refractive index of Ge measures as high as 4 in the Mid-IR region, making it a suitable material for practical chemical sensing, where longer wavelengths and compact sensor designs are required. Numerous research efforts have been put into the germanium-on-silicon platform (GOS) as it is straightforward to deposit Ge onto the Si substrate, and also because these two materials have a wide transparency range. Chang *et al.* first developed a Ge strip waveguide on the GOS platform [34]. This platform was a 2 μm -thick monocrystalline germanium layer on a silicon substrate, which was covered with a 10 μm -thick intrinsic silicon buffer layer. The Ge layer was grown with reduced pressure chemical vapor deposition (RPCVD), and the strain in the Ge film was completely relaxed. The size of the strip waveguide was 2.9 μm wide and 2 μm thick, with the propagation loss measuring 2.5 dB/cm at the wavelength of 5.8 μm in TM mode polarization, and the bending loss measuring 0.12 dB for a 90° bend with a radius of 115 μm . The SEM image of a fabricated strip waveguide with a taper coupler is illustrated in *Figure 11*.

Figure 11. SEM image of the waveguide with a wide taper coupler. The inset is the cross-section in the straight section of the waveguide [34].

The most impressive results were gathered by Mashanovitch *et al.*, showing the waveguide loss to be as low as 0.58 dB/cm at 3.8 μm wavelength [35]. The low propagation loss was realized by applying a fine-designed rib waveguide geometry, instead of the conventional strip waveguide. It is well-known that the lattice constants of Ge and Si are different, resulting in misfit dislocation defects at the Ge/Si interlayer during the Ge deposition process, as shown in *Figure 12(a)*. These defects will cause scattering loss when light is transmitted in the waveguide. In order to avoid this problem, the researchers designed a rib waveguide structure on the GOS platform to elevate the guiding mode from the bottom of the waveguide, eliminating contact between the guiding mode and the defects. This structure is shown in *Figure 12*. The dimension of the rib waveguide is 2.9 μm thick and 2.7 μm wide, with an etch depth of 1.7 μm . After optimizing the fabrication process, they obtained the waveguide loss of 0.58 dB/cm; the lowest result published at the Mid-IR range thus far.

Figure 12. (a) Transmission Electron Microscope (TEM) image of the Ge-on-Si interface region, showing that most dislocations are located at this interface. (b) SEM image of a cleaved Ge-on-Si rib waveguide cross-section [35].

Nedeljkovic *et al.* took these results a step further and investigated these devices at even longer wavelengths ($7.5\text{-}8.5\ \mu\text{m}$) [36], reporting the transmission measurements of GOS rib waveguides with minimum propagation losses of $2.5\ \text{dB/cm}$ at $7.575\ \mu\text{m}$, but showing increasing losses at higher wavelengths which were left unexplained. As the transmission wavelength increases, the dimension of the rib waveguide would also change correspondingly. According to their simulation results, the $3\text{-}\mu\text{m}$ -thick Ge layer with $1.8\ \mu\text{m}$ etching depth could still support the single TE mode, but the width was required to increase to $4.3\ \mu\text{m}$.

Besides the Ge-on-Si platform, a waveguide platform based on SiGe graded index material, cladded by a thick epitaxial Si layer, was proposed by Brun *et al.* [37]. The advantages of the SiGe graded material on Si substrate include full band covering from $3\text{-}8\ \mu\text{m}$ and the limiting of diffusion losses in the perpendicular direction via the use of an interface-free graded index stack. The thick SiGe core layer was deposited by RPCVD and the ratio between germane and dichlorosilane was gradually modified during growth, in order to change the relative concentration of Ge. The Ge content was controlled with an approximately linear increase of up to 40% in the first half of the profiled layer, and linearly decreased back to 0% in the second half. Straight

waveguides showed losses as low as 1 dB/cm at the wavelength of 4.5 μm , and 2 dB/cm at 7.4 μm , with different widths of the waveguides.

Figure 13. SEM image of a SiGe waveguide core after etching of the waveguide core layer. In the inset: a cross section of the final structure showing the SiGe core completely encapsulated with the epitaxial Si cladding layer. Intensity grading in the vertical direction is related to the variation of the Ge concentration [37].

The same group of researchers also developed a new platform based on a Ge core embedded in thick $\text{Si}_{80}\text{Ge}_{20}$ layers [38]. The SEM images of the Ge strip waveguide, before and after being cladded with the thick SiGe layer, are shown in *Figure 14*.

Figure 14. Cross section of a Ge/SiGe waveguide before and after SiGe cladding [38].

The refractive index of the SiGe alloy can be determined by $n_{\text{Si}(1-x)\text{Ge}x} = x n_{\text{Ge}} + (1-x) n_{\text{Si}}$, where n_{Ge} and n_{Si} are the refractive indices of Ge and Si respectively, and x the percentage content of Ge in the SiGe alloy. Thus, the index difference between the core and the cladding is around 0.5, over the spectral range of 3-13 μm . The core has a

typical cross section of $2.5 \times 2.5 \mu\text{m}$ and is capped with a $4\text{-}\mu\text{m}$ -thick SiGe cladding layer. The propagation loss given by the waveguide spirals was 3.5dB/cm at the wavelength of $8 \mu\text{m}$. Though this platform is suitable to accommodate devices spanning over more than an octave, the index contrast is low; whereas the devices are generally comparatively large.

Similar to the SOI structure, germanium-on-silicon dioxide (GOI) has been proposed for Mid-IR applications, because of the wide transparency range of Ge as well as the ease of removing the buried oxide layer by a simple wet etching process. Jian Kang *et al.* has fabricated GOI wafers via the smart-cut technique and aluminum oxide (Al_2O_3) bonding interface [39].

Figure 15. (a) Cross sectional SEM image of the bonded GOI wafer. (b) Tilted top view of fabricated rib waveguide on GOI [39].

First, a SiO_2 capping layer was deposited on a bulk Ge wafer by PECVD. After hydrogen ion implantation on the Ge surface, the SiO_2 layer was removed by wet etching. Then, a thin Al_2O_3 layer was deposited on the Ge surface, which served as half of the bonding interface layer. Meanwhile, a thin Al_2O_3 layer with the same thickness was also deposited on a Si handle wafer, upon which a $2\text{-}\mu\text{m}$ -thick SiO_2 layer was thermally grown in advance. Then, the Ge wafer was flipped over and bonded onto the Si handle wafer, followed by annealing to split the bulk Ge. After bonding, the Ge surface was polished by chemical mechanical polishing to reduce surface roughness. Rib waveguides were fabricated on this platform with a thickness of 300 nm , slab thickness of 100 nm , and a width of $2 \mu\text{m}$. The rib waveguides were characterized at the

wavelength of $1.95\ \mu\text{m}$, with a loss of 1.4 dB/mm. Based on the GOI platform, the suspended Ge membrane platform was realized by removing the buried oxide layer. The photonic crystal cavity, photonic crystal waveguide, suspended membrane waveguide, and focusing sub-wavelength grating coupler have been fabricated on this platform by Xiao *et al.* [40]. The waveguide loss was not reported, but accounting for the quality of the etching process and the low surficial roughness of the Ge structures, good performance up to the higher end of the Mid-IR range can be expected.

Besides the conventional materials like SiO_2 or SiN_x used for the under-cladding layer, the possibility of using other materials has been explored by Kim *et al.* who reported a new Ge waveguide platform on F- and Y-based insulator cladding, such as CaF_2 and Y_2O_3 . These materials have a small refractive index, broad transparency range, and high thermal conductivity [41]. This new Ge-on-insulator platform was realized by wafer bonding and splitting. First, a thick Y_2O_3 layer was deposited on a commercial GOI wafer and a Si handle wafer respectively. After surface treatments, the two wafers were bonded together and the Si substrate of the GOI wafer was split by removing the BOX layer with HF-based solution. The bonded wafer is shown in *Figure 16*, but the experimental results of propagation losses are not reported in the paper; only the simulation results are shown.

Figure 16. Cross section SEM of the bonded Ge-on-Y2O3 structure [41].

2.1.3 Dielectric and Chalcogenide Based Platform

The advantage of using dielectric materials for the core and cladding layer is the ease of fabrication by chemical vapor deposition, in which no smart-cut, wafer bonding or layer transfer is required. In ref [42], a SiN_x core waveguide upon SiO_2 tips for glucose sensing was demonstrated. A strip SiN_x waveguide with a thickness of $2\ \mu\text{m}$ and a width of $10\ \mu\text{m}$ was etched on a thick thermal SiO_2 layer, which was later etched by HF to form a pedestal shape with a thickness of $5\ \mu\text{m}$ and a notch width of $2\ \mu\text{m}$. The losses were measured to be $0.16\ \text{dB/cm}$ at $\lambda=2.7\text{-}2.81\ \mu\text{m}$ and $2.1\ \text{dB/cm}$ at $\lambda=3.5\text{-}3.7\ \mu\text{m}$. Besides SiN_x , a $1\text{-}\mu\text{m}$ -thick aluminum nitride waveguide on $3\text{-}\mu\text{m}$ -thick thermal oxide, with a propagation loss of $0.83\ \text{dB/cm}$ at the wavelength of $2.5\ \mu\text{m}$, was reported in ref [43]. Its main limitation was the operation wavelength range, which is related to the absorption of the material used.

Figure 17. SEM image of the fabricated SiN_x waveguide [42].

Conversely, chalcogenide materials offer potential for long wavelength operations, and the fabrication of a high-index chalcogenide waveguide on top of a lower-index chalcogenide layer has been successfully realized with low loss of $2.5\ \text{dB/cm}$, at $7.7\ \mu\text{m}$ wavelength [44-46]. However, chalcogenides are very fragile and their compatibility within CMOS foundries has yet to be fully validated.

2.1.4 Summary

To summarize, tremendous efforts have been put into the study of Mid-IR integrated photonic technologies, due to the rising demand of chemical and biological sensing. The maturity of the technologies developed for data communications, focusing on the wavelengths of 1.3 and 1.55 μm , has helped the deployment of related integrated photonics toward longer wavelengths (2-15 μm), such as the well-established SOI platform and foundry processing. Besides the SOI platform, many novel platforms have been proposed and demonstrated, based on two main tendencies with larger transparency windows, such as SON, SOS, GOS, and the SiGe alloy; and the realization of air-suspended structures which use either Si or Ge as the core material, by removing the buried oxide layer. Even if the suspended structure is conceptually realizable and technologically simple, its scalability, intrinsic robustness, and the reliability of a platform based on this structure are still in question. The table below summarizes the platforms that have been used for Mid-IR photonics.

Wavelength	2-4 μm	4-8 μm	8-15 μm	References
Si/SiO ₂	3@3.8 μm 1.5@3.8 μm 1.28@3.8 μm 1.4@3.8 μm			[6] [7] [7] [8]
Si/Al ₂ O ₃		4.3@4.5 μm 1.92@5.18 μm		[10] [11]
Si/SiN _x	5.1@3.39 μm			[9]
Si/Si Pedestal	2.7@3.7 μm			[12]
Si/SiO ₂ Pedestal	Not reported	Not reported	Not reported	[13]
Suspended-Si	0.82@3.8 μm 2.8@2.25 μm	3.1@7.67 μm		[14][15] [16]
SiGe/Si		1@4.5 μm	2-3@5.5-8.5 μm	[21] [47]

		2@7.4 μm		[21]
Ge/SiGe			3.5@8 μm	[22]
Ge/Si	0.58@3.8 μm	2.5@5.8 μm 2.5@7.58 μm	1@10 μm	[19][18] [48] [20]
Ge/SiO ₂	1.4@1.95 μm			[23]
Suspended-Ge	Not reported	Not reported	Not reported	
Ge/chalcogenide	Not reported	Not reported	Not reported	
SiN/SiO ₂	0.16@2.7-2.81 μm 2.1@3.5-3.7 μm			[26] [26]
AlN/SiO ₂	0.83@2.5 μm			[27]
Chalco/chalcogenide		2.5@7.7 μm		[28]

Table 1. Passive Waveguide Platforms Performance in dB/cm for the Mid-IR (Core/Bottom cladding if Any)

2.2 Platforms Realized by Wafer Bonding

Wafer bonding has drawn much research interest, due to its capabilities in heterogeneous integration of materials, 3D integration and packing, and microelectronicmechanical (MEMS) systems application. It is able to realize new designs and combinations of different materials and structures, which was initially ruled out by conventional material science and semiconductor processing technology [49]. Therefore, wafer bonding can be applied in group IV photonics devices, sensors, and various other research fields, such as power devices, III-V compound-based devices etc. [50].

The wafer bonding process was first proposed in 1983 by Kimura *et al.* [51]. In this paper, they described the fabrication process of the SOI wafer by using wafer bonding and etch-back technology. They successfully bonded two Si wafers together, with a glass layer coating on each wafer, to form the SOI structure. Two types of Si substrates were prepared in their process. A boron-doped Si wafer was used as a p^+ -Si substrate, followed by epitaxial growth made at 1050°C without intentional doping, by a conventional CVD method using SiH₄ gas. Then, the epitaxial Si film surface was

oxidized at 950°C in wet O₂ for 30 min after epitaxial growth; after which SiN_x and SiO₂ films were deposited on the oxidized epitaxial Si film surface by CVD. Another Si wafer, called the Si handle wafer, with an orientation of <511>, was used for ease of evaluation of the crystalline quality of (100) epitaxial Si film following transfer. The Si handle wafer was oxidized at 1100°C in wet O₂ and 0.6 μm SiO₂ was formed. Glass of the PbO-B₂O₃-SiO₂ type was used for bonding these two substrates. An organic solution involving both the glass powder and ethylcellulose was prepared and coated on each substrate surface type, using a conventional spinner. Then, two wafers with a 3 μm glass layer on each surface were contacted with the weight of a fused-silica block, which were then heated up to 930°C for 10 min, followed by a cooling-down period. Finally, the p⁺-Si substrate was polished down to 30 μm, followed by selective etching with HF-HNO₃-HAc, which is commonly termed the etch-back process. The bonding process is complicated, as many materials, as well as both oxidization and deposition processes, are involved. The crystalline quality of the final bonded SOI was lower compared to the original epitaxial grown Si film, but better than that of silicon on sapphire [52], which is another SOI electronic material typically used for integrated circuit fabrications. Lasky *et al.* developed a method for wafer bonding without the help of any glue or glass insertion layer [53]. Their process consists of growing a lightly-doped Si epitaxial layer on a heavily-doped Si substrate, then growing a thermal oxide which was bonded to a second, lightly-doped Si handle wafer. A preferential etch [54] was used to remove the heavily-doped substrate, leaving the thin, lightly-doped epitaxial layer above the thermally grown oxide. They claimed a pivotal improvement of their process compared with previous bonding and etch-back processes [51]. Their bonding method allows thermally grown oxides on the two wafers to be bonded over the entire area of the wafer, with no degradation of the quality of Si thin films. Bonding was achieved by simply pressing the oxidized surfaces of the two wafers together and inserting them into an oxidizing atmosphere at higher than 700 °C. At such a high temperature, the gaseous oxygen trapped between wafer pairs was converted to silicon dioxide, after which a partial vacuum was created, forcing the wafers into intimate

contact. Once the wafers were pressed into intimate contact, the bonding chemical reaction is believed to happen as follows:

The researchers claimed that the bonding strength was strong, and that the quality of the Si layer on the bonded substrate was not degraded from its original bulk quality, based on electrical characterization using capacitor structures, as well as n-channel and p-channel transistors fabricated on the bonded SOI wafer. The photograph of the bonded SOI under monochromatic light, as well as the cross-sectional TEM image after etching, are shown in *Figure 18*.

Figure 18. (a) The bonded SOI wafer under monochromatic light. (b) cross-sectional TEM of the SOI after etching to form Islands [51].

Besides the wafer bonding technology, the layer transfer technique is also critical to realize the required structure, for example, single crystalline semiconductor materials on top of an insulator layer. This transfers a certain layer from a donor wafer to a handle wafer to realize hetero-structures, such as Si on quartz wafer, SiGe on insulator, strained Si on insulator and Ge on insulator (GOI), as well as to preserve the crystal quality, optical, and electrical properties at the same time [55-57]. Thus, the layer transfer technique is important to wafer bonding and the realization of high-quality hetero-structures. Layer transfer, realized by the Smart Cut technique, was first introduced in 1995 for SOI fabrication [58]. This process provides a high-quality SOI

wafer that can meet the criteria necessary for the development of ultra large-scale integration technologies: thickness homogeneity, good crystalline quality, low cost, and availability. The Smart Cut process basically comprises the four main steps illustrated in *Figure 19*.

Figure 19. Principle of the Smart-Cut process [56].

The first step is hydrogen implantation, into a wafer-A capped with a dielectric layer. The second step is the hydrophilic bonding of wafer-A at room temperature, to a handle wafer-B. Both wafers are cleaned beforehand with RCA solution. The third step is the two-phase heat treatment of the two bonded wafers. During the first phase (400-600 °C), the implanted wafer A splits into two parts: a thin layer of monocrystalline silicon remaining bonded to wafer B, which gives rise to a SOI structure; and the remainder of wafer A. A second high temperature treatment phase (around 1100 °C) strengthens the chemical bonds. The final step involves polishing the surface after splitting. The Smart Cut technique provides a versatile way to obtain high quality SOI, as well as numerous heterostructures like GOI. This technique is considered the best way to form wafer-

level GOI. However, the mechanical and thermal properties of Ge pose size constraints in GOI fabrication, whereby fabrication of 300mm and above via Smart Cut becomes difficult and costly. Moreover, the thermal mismatch of the heterostructure increases with wafer size, which further restricts the application of the Smart Cut technique in GOI fabrication.

Many platforms used for Mid-IR photonics are realized by wafer bonding and layer transfer techniques. The SON platform mentioned above is fabricated with this technique, and its schematic fabrication flow is demonstrated in *Figure 20* and described in the following. First, a SOI wafer and a Si handle wafer were diced into small pieces ($2 \times 2 \text{ cm}^2$). The SOI die was then coated with $1.3 \text{ }\mu\text{m}$ -thick low-stress SiN_x layer, using plasma enhanced chemical vapor deposition (PECVD). After that, SiO_2 was deposited on top of the SiN_x layer and Si handling wafer respectively, to enable the bonding process by PECVD. The authors in ref [25] chose a spin-on-glass (SOG) layer as the bonding layer because of its low cost and ease of fabrication. Since SOG has low adhesion to Si and SiN_x surfaces, SiO_2 was first deposited on the SOI and Si handle wafer. Then, the SOI die was flipped over and bonded to the Si handling die, after which the bonded piece was sent into a chamber for annealing to enhance bonding strength. Finally, the Si substrate and the BOX layer of the SOI die were removed by lapping and diluted HF wet etching to expose the thin Si film for waveguide processing. Instead of using wafer-to-wafer bonding, the authors in ref [25] used small diced pieces for die-to-die bonding to realize the silicon-on-nitride structure, which makes bonding easier with a higher rate of success; but the low throughput of die-to-die bonding may not be suitable for the CMOS foundry process. The application of SOG as the bonding layer also introduced another spin coating process, which the authors did not mention if such material was compatible with the CMOS process.

Figure 20. Schematic of silicon-on-nitride die fabrication process [25].

Another platform, that has been extensively studied for electronics and photonics applications on CMOS integrated circuits, as well as photonics integrated circuits, is the GOI platform. In ref [23], the authors reported a waveguide with a propagation loss of 1.4 dB/cm at the wavelength of $1.95 \mu\text{m}$ on the GOI platform, which was realized by the smart-cut process with Al_2O_3 as the bonding layer. Instead of using the smart-cut process, GOI also can be realized by direct wafer bonding and the layer transfer technique. Lee *et al.* demonstrated the GOI fabrication process in their paper [59], in order to provide a virtual substrate for III-V material epitaxy growth. The fabrication flow of the GOI wafer is illustrated in Figure 21 and described as follows. A $1.2\text{-}\mu\text{m}$ -thick Ge layer was deposited on an 8-inch Si substrate in a metal-organic-chemical vapor deposition (MOCVD) system. The Ge-on-Si substrate served as a donor wafer in the wafer bonding processes. After Ge deposition, the donor wafer was cleaned by chemicals to remove organic contaminates and to dissolve germanium oxide. Then, the donor wafer was bonded to a Si handle wafer with a thermal oxide layer on top. Before wafer bonding, oxygen plasma was applied to the surfaces of the Ge layer and thermal oxide layer to enhance surface hydrophilicity. Then, the donor wafer was flipped over and bonded onto the thermal oxide layer of the handle wafer at atmospheric pressure by pressing the bonded wafer pair. After the wafer bonding, a low temperature annealing of 300°C in ambient N_2 was carried out for several hours to enhance bonding strength. Then, the Si donor substrate was removed, by grinding and wet etching in tetramethylammonium hydroxide (TMAH), which has a much higher etching rate to Si

than Ge. The researchers also investigated the influence of the thickness of the thermal oxide layer on bonding quality, and demonstrated that a thicker thermal oxide layer can absorb more H₂, thus reducing thermal bubbles during post-bonding annealing.

Figure 21. Process flow of obtaining GOI wafer by wafer bonding, layer transfer, etch back, and smoothening [57].

The wafer bonding and layer transfer process provides an approach to remove the Si donor substrate, in order to expose the Ge/Si interfacial layer, where misfit dislocation defects exist. Therefore, a high-quality Ge device layer can be achieved by removing this defective interfacial layer. Another advantage of the wafer bonding and layer transfer process is that it provides a more convenient and flexible method for doping the Ge device layer, to realize a vertical *p-i-n* structure for active devices, such as modulators or photodetectors. Lin *et al.* reported high-efficiency normal-incidence vertical *p-i-n* photodetectors, on a GOI platform that was fabricated with the same process as described in ref [41]. The doping process was completed along with the bonding process. After Ge deposition on the Si donor wafer and before bonding, boron ions were implanted into the top surface of the Ge layer. After bonding and the removal of the Si donor substrate, arsenic ions were implanted into the other surface of the Ge layer, which was initially at the Ge/Si interface. The wafer bonding and layer transfer technique introduces a viable approach to realize both *p*⁺- and *n*⁺-Ge layers, and also

eliminates the concern of excess diffusion of *in situ*-introduced dopants during Ge growth and cyclic annealing [60]. A recently-reported Ge-on-insulator platform for Mid-IR photonics, Ge-on-Y₂O₃, was realized by the wafer bonding and splitting process [41]. Y₂O₃ was chosen to be the under-cladding material because it has a small refractive index, broad transparency range, and high thermal conductivity. These advantages of this material make it a suitable candidate for Mid-IR sensing application and light-emitting devices, especially lasers, as its broad transparency range can cover broader fingerprint regions of molecules and its small refractive index can form a large refractive index contrast with the core Ge layer, thus ensuring a sharp bent waveguide design feasible. For lasers, the Y₂O₃ under-cladding can be a feasible heat sink, due to its high thermal conductivity, compared with other insulators like SiO₂. A commercial GOI substrate and a Si handle wafer were involved in the fabrication process. First, the commercial GOI wafer was dipped in a diluted HF solution to remove the native oxide. Then, Y₂O₃ was deposited on both the GOI and Si handle wafers by sputtering, for thick Y₂O₃ deposition. After the deposition of thick Y₂O₃, to make the surface smooth enough to initiate bonding, CMP was carried out, followed by surface treatments like O₂ plasma irradiation to guarantee good bonding behavior. Subsequently, the two wafers were brought together with the treated Y₂O₃ layers facing each other. Finally, the BOX and Si substrates of the GOI wafer were split by selective etching, using a HF-based solution, as only SiO₂ is etched by the HF-based solution, whereas Y₂O₃ is not affected. The process completed the transfer of the Ge layer to another Si handle wafer, with Y₂O₃ as the under-cladding. The schematic of the fabrication process is shown in *Figure 22*.

Figure 22. Fabrication flow of Ge-on-Y₂O₃ using wafer bonding and layer splitting [41].

Many platforms designed for Mid-IR photonics are realized by wafer bonding, because of the demand for replacing or inserting a different insulator as the under-cladding layer, to realize a large refractive index contrast between the core material and the under-cladding. This extends the transparency range, as well as provides a high thermal conductivity. The main method to realize high-quality single crystal Group IV materials on different insulators is, namely, wafer bonding. Its operation wavelength can be extended to 15 μm by using the new platforms realized by the wafer bonding technique. The operation wavelength of each platform is detailed in the table below. The bonding process and the materials involved are entirely CMOS-compatible, which makes the integration of electronics and photonics possible. Thus, the wafer bonding and layer transfer technique is a very critical process for Mid-IR photonics applications.

Platforms	Fabrication Process	Bonding Layer	Operation Wavelength	Reference
SOI	Wafer bonding and etching back	Glass	<3.8 μm	[33]
SOI	Wafer bonding and	SiO ₂	<3.8 μm	[35]

	etching back			
SOI	Smart-cut	SiO ₂	<3.8 μm	[40]
SON	Wafer bonding and layer transfer	SOG	2~9 μm	[9]
GOI	Wafer bonding and layer transfer	SiO ₂	>3.8 μm	[41]
Ge-on-Y ₂ O ₃	Wafer bonding and layer splitting	Y ₂ O ₃	2~15 μm	[25]

Table 2. The fabrication method, bonding layer, and operation wavelength of the platforms for Mid-IR photonics.

2.3 Mid-IR Sensing Application

Group IV photonics has been intensively studied in the recent years, due to its compatibility with silicon processes, small footprint, low fabrication cost, immunity to electromagnetic interference, as well as its sensing applications in the Mid-IR range [16]. In particular, the Mid-IR spectrum represents a primary spectral range for photonic sensing applications, as the characteristic absorption fingerprints of most molecules reside in it. Waveguide sensors are especially useful in Mid-IR because of their relative simplicity and accuracy. They are based on the interaction of the evanescent field of a waveguide mode with the surrounding media, and the resulting perturbation in the intensity of the mode at the output end of the waveguide. These devices provide a promising method for the label-free detection of target molecules, as well as for the real-time monitoring of solutions, gases, or reactions that occur near the device surface. Unlike the conventional Fourier transform infrared spectroscopy (FTIR) that is limited to usage in laboratories due to its large size and the need of specific knowledge to understand the data, these devices are small in size and require a minimal sample test volume. The reported Mid-IR strip waveguides for laser spectroscopy have 1000 times higher sensitivity than FTIR in the liquid environment [61]. Thus, these devices will have a major impact on environment studies (e.g., detection of hazardous

and greenhouse gases), industrial leak detection, process control, food processing, medical breath analysis, etc. [62].

Another sensing mechanism is based on resonance peak shifts of optical cavities, caused by the change in the background refractive index [63-66]. This change in refractive index value can be introduced by either the appearance of target molecules or a change in the concentration of certain substances. However, this mechanism is mainly limited by laser frequency noise, and the direct interaction between analytes and cavities will cause contamination to the cavities, thus limiting cycle timings [67].

Due to the relative simplicity and effectiveness of waveguide sensors, based on evanescent wave sensing using a mid-infrared light source as the probing light, many waveguide sensors on diversified platforms aiming for different target analytes have been demonstrated, such as chalcogenide thin film, GOS, SOI, and SOS. Chalcogenide has a wide transparent window in the Mid-IR range and a tunable refractive index, which makes it a suitable material for Mid-IR sensors, based on evanescent field absorption. Many research results on Chalcogenide waveguide sensors have been reported [68, 69]. However, due to its fragileness and incompatibility with the CMOS fabrication process, Chalcogenide may not form a good candidate for Mid-IR waveguide sensors in photonic integrated circuit applications. A single mode strip waveguide on the Ge-on-Si platform was used as a sensor for cocaine detection by Chang *et al.* [11]. The GOS platform was selected because Ge and Si have low absorption over a wide range of Mid-IR wavelengths. A single mode strip waveguide was designed and fabricated with thickness of 2 μm and width of 2.9 μm at the wavelength of 5.8 μm , where the absorption peak of cocaine resides. The strip waveguide has a higher fraction of energy in the evanescent field compared to a slab waveguide, hence it displays a higher sensitivity. A microfluidic chamber integrated with an inlet and an outlet for liquid handling was bonded onto the waveguide chip. The schematic of the sensor chip and cross-sectional SEM of the Ge waveguide on Si are illustrated in *Figure 23*. The lowest concentration detected was 100 $\mu\text{g/ml}$.

Additionally, the researchers claimed that the detection limit can be further improved by reducing system noise and using a longer waveguide.

Figure 23. (a) Schematic of the waveguide sensor bonded with the microfluidic chamber. (b) SEM image of the cross-section of the Ge strip waveguide on Si [11].

Lin *et al.* also studied the possibility of using the Si waveguide as a Mid-IR sensor for a lab-on-chip spectrometer, as crystalline Si is highly transparent between 1.3 to 6.5 μm , such that its operational spectral range covers most characteristic chemical absorption bands, due to the vibration of chemical bonds such as C-H, N-H, and O-H [70]. Since the SOI platform was used, a pedestal waveguide structure was designed to avoid the absorption of the buried oxide beyond 3.8 μm . The geometry of the pedestal waveguide is shown in Figure 24 and the parameters w , h , s , and d denote the waveguide width, waveguide height, pedestal height, and the notch width, respectively, which were set to be 8 μm , 5 μm , 14 μm , and 2 μm respectively for single mode transmission.

Figure 24. (a) Schematic of pedestal waveguide and its parameters. (b) The SEM image of the fabricated waveguide [68].

The researchers successfully demonstrated *in situ* identification of chemical compositions and concentrations of organic solvents. The Mid-IR sensor is able to distinguish hexane from the rest of the tested analytes, since hexane has a strong absorption from the aliphatic C-H stretch at the wavelength of $3.55\ \mu\text{m}$. When applying the wavelength of $3.3\ \mu\text{m}$, the Mid-IR sensor was also able to detect the concentration of toluene dissolved in carbon tetrachloride, by monitoring the change of the output intensity of light, because toluene has a strong absorption at the wavelength of $3.3\ \mu\text{m}$ from the aromatic C-H stretch, and this absorption increases as the concentration of toluene increases. The sensing results are shown in *Figure 25*.

Figure 25. (a) The waveguide mode intensities at different toluene concentrations. (b) Hexane shows a large difference in absorbance compared to other analytes [68].

The researchers also demonstrated a slot waveguide for the Mid-IR spectrometer, claiming that its sensitivity can be increased by 50 times compared to normal waveguides, as the special geometry of the slot waveguide can confine most of the light in the low-refractive index gap, where the light can directly interact with analytes. This spectrometer accurately distinguished n-bromohexane, toluene, and isopropanol; and determined the ratio of the mixture of acetonitrile in ethanol, at a low concentration smaller than $5\ \mu\text{l/ml}$ [71].

A numerical analysis of the waveguide sensor on the silicon-on-sapphire platform at Mid-IR was reported by Huang *et al.* [72]. Three different types of waveguide were investigated on their geometrical dependence on evanescent field fraction and propagation loss, which have critical influence on sensitivity. They took the detection of CO₂ in the atmosphere, based on its Mid-IR absorption peak at around $4.23\ \mu\text{m}$, as

an example. Unfortunately, they did not report such experimental data in their paper. Singh *et al.* demonstrated the Mid-IR absorption sensing of heavy water using a silicon-on-sapphire waveguide [73]. Heavy water has a molecular weight 11% higher than regular water, which is the biggest weight difference between any atom and its stable isotope. This property makes it useful for various fields; for example, it can be used for increasing the photon emission of fluorescent proteins in bio-imaging. Since heavy water is toxic at high concentrations, measuring its safe level in biological systems using a miniaturized device would be helpful [74]. The researchers designed and fabricated strip waveguide sensors on the SOS platform, with a total length of 0.52 cm. The propagation loss of the waveguide was 1 dB/cm; while after the presence of silica upper-cladding, this loss increased to 13 dB/cm at the wavelength of 4 μm , with the total insertion loss of the SOS waveguide being 12 dB. A light source with 4 μm wavelength was used, because the absorption coefficients of heavy water and regular water has a large disparity of between 18000 cm^{-1} and 144 cm^{-1} respectively, at this wavelength. Then, heavy water and regular water were mixed with different concentrations of heavy water, followed by a droplet test. Droplets with different concentrations were dropped onto the chip to fully cover the waveguide, and the transmissions at the output end of the waveguide were recorded. The lowest concentration measured 0.25% heavy water in regular water, which translates to 2500ppm. The layout of the sensor and the measurement result are shown in *Figure 26* below.

Figure 26. (a) Layout of the SOS sensor cladded with the sample liquid. Inset: picture of the SOS chip with a water mixture. (b) Normalized relative transmission of the waveguide at different water mixtures [71].

The performance of the waveguide sensors on different platforms are reviewed in this section. Analytes such as cocaine, heavy water, toluene, and hexane were detected by the waveguide sensors to demonstrate that the Mid-IR waveguide sensor can be applied to biochemical and health detection (e.g. drug detection) and environment monitoring (e.g. hazards or air pollution detection). The main concern with the Mid-IR waveguide sensor is how to improve its sensitivity and the suitability of integration with electronic and photonic components.

Chapter 3 Fabrication and Characterization of the Engineered GON Wafer

3.1 Introduction

Many efforts have been put into new material systems to extend the operation wavelength of Group IV photonics into the Mid-IR range, due to its promising applications in spectroscopy, biochemical sensing, environment monitoring, and free space communication. The standard silicon-on-insulator waveguides are unsuitable for Mid-IR, particularly above the $3.6\ \mu\text{m}$ mark, since the material loss of the buried oxide layer becomes substantially high. Many alternative material systems have been proposed and studied, namely silicon on chalcogenide, silicon on sapphire, silicon on nitride, and germanium on silicon, as mentioned earlier. These platforms can realize the extension of the operation wavelength up to the Mid-IR range; however, the core/clad index contrasts are not as large as that of SOI, which makes small radii of bends impossible to achieve. Thus, a new material system that can operate in the Mid-IR range, with also a large refractive index contrast between core and claddings, is required. Hence, the germanium-on-silicon nitride (GON) structure was proposed and realized. SiN_x is transparent up to $7\ \mu\text{m}$ [16] and has a refractive index as small as 1.9, as indicated by the results of ellipsometer measurements. However, making a GON wafer is not as straightforward as depositing a Ge layer onto the SiN_x layer. Because SiN_x is amorphous, it is impossible to get high-quality single crystal Ge by direct deposition. Therefore, a way to realize a high-quality Ge layer on SiN_x under-cladding is required. This can be done by wafer bonding and layer transfer techniques. First, a high-quality Ge-on-Si wafer with low threading dislocation density (TDD) is produced. Then, the SiN_x layer is deposited on top of the Ge layer. After the deposition of SiN_x , the wafer is flipped over and bonded to another Si handle wafer, followed by removing the Si donor wafer. After removing the Si donor wafer, the initial defective interface between Ge and Si is now exposed on top of the wafer. This defective surface may degrade the performance of Ge waveguides, causing high propagation losses. Nonetheless, the top surface can be chemical mechanically polished to obtain a flat and

smooth surface, ensuring the performance of Ge waveguides. Now, the preparation work and detailed fabrication process of the GON wafer will be introduced in this chapter.

3.2 Bonding Criterion

It is only under the condition that critical requirements in bonding energy, surface cleanness, roughness, and flatness can be satisfied, that successful wafer bonding is achieved. Bonding at room temperature is realized through the formation of hydrogen bridge bonds or Van Der Waals interactions. Normally, it is required that a force be applied at one point of the bonding pair to allow the bond front to propagate. Whether the bond front can propagate and lead to successful bonding is determined by the balance between surface energy dissipated, due to bond formation, and the strain energy required to deform the wafers into a common shape [75]. The surface energy dissipated during the bond formation, also known as the Dupré work of adhesion (W), is shown in *Figure 27(a)*. W can be expressed as:

$$W = \gamma_1 + \gamma_2 - \gamma_{12} \quad (2)$$

where γ_1 , γ_2 are the surface energies of two solids, and γ_{12} is the interface energy.

Figure 27. (a) The force separation curve for two surfaces. The grey area represents the work of adhesion. (b) The change in system energy as two surfaces are bonded. Surface energy (γ_1 , γ_2) is lost, interface energy (γ_{12}) and strain energy (U_E) are gained [75].

As shown in *Figure 27(b)*, the total energy of the bonding system U_T can be expressed as:

$$U_T = U_E + aB\gamma_{12} + (L-a)B(\gamma_1 + \gamma_2) \quad (3)$$

where U_E is the elastic strain energy, a the bond front position, and B the structure width. The bond front stops propagating when the system is in equilibrium. This case can also be described as $dU_T/da=0$. Combining equation (2) and (3), the equilibrium condition can be written as:

$$\frac{1}{B} \frac{dU_E}{da} = W \quad (4)$$

This can be generalized to three dimensional situations, by taking the derivative of the strain energy with respect to the area of the interface through bonding. Since the bond front will propagate until the equilibrium condition is satisfied, the bonding criterion can be expressed as:

$$\frac{dU_E}{da} \leq W \quad (5)$$

The surface cleanness of the bonding pair is critical for direct wafer bonding, which affects the structural, electrical, and optical properties of the bonding interface, as well as the bonded wafers. This surface needs to be free of organic and metallic contaminations and particles; thus, proper surface cleaning needs to be applied to treat the wafer surface before direct wafer bonding. In addition, the materials and processes used for cleaning have to ensure not to degrade the surface or cause severe surface roughening. The cleaning method used in semiconductor industry, Radio Corporation of America (RCA) cleaning, can also be used in the direct wafer bonding surface treatment. The RCA cleaning method involves two hydrogen peroxide-based steps: RCA1 ($\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O} = 1:1:5$) and RCA2 ($\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O} = 1:1:6$). RCA1 is used to remove organic contaminants, especially hydrocarbons, through the oxidizing action of H_2O_2 and the solvating action of NH_4OH . Conversely, RCA2 is designed to remove metallic or ionic contaminants. Since ammonia from RCA1 can attack the surface and increase surface roughness, reducing the ratio of NH_4OH is suggested for surface cleaning in wafer bonding. Besides metallic and organic contaminants, the presence of particles will also have a large influence on the quality of direct wafer bonding. The

particles function as spacers and prevent intimate contact between two mating wafers, leading to unbonded areas or intrinsic voids. The sizes of particles and bonding strength determine the size of the void. Assuming the size of particle (radius h) is much smaller than the thickness of the wafer d , and the radius R of the resulting unbonded circular area is much larger than d , as illustrated in *Figure 28(a)*, an approximation of the radius of the void can be expressed as follows, assuming the particle to be incompressible [50].

$$R = (1.2Ed^3 / \gamma)^{1/4} h^{1/2} \quad (6)$$

where E is Young's modulus, and γ is the specific interface or surface energy of the bonding interface (about 100 erg/cm² for hydrophilic Si/Si bonding at room temperature). Even for sufficiently small particles, the resulting void is large compared to the size of the particle. The size of the void decreases dramatically with a reduction in the dimensions of the particle. If the particle size is small enough, the resultant void size is of a much smaller radius, as shown in *Figure 28(b)*, which can be written as;

$$R = kh \quad (7)$$

where k is a dimensionless factor.

Figure 28. (a) Schematic of particle leading to an unbonded area with a radius R larger than the thickness of wafer d . (b) the same for sufficiently small particles resulting in R much smaller than d [49].

Since particles with sizes as small as a few micrometers can result in voids of several millimeters, particle control is essential to void-free bonding. In order to reach the particle-free bonding requirement, some solutions can be applied, such as the use of improved cleanroom environments like the Class 1 cleanroom, megasonic cleaning,

and dust-free transport of wafers. Usually, cleanrooms in research institutions do not meet the Class 1 standard, thus the pre-bonding cleaning process and dust-free transport between different bonding equipment become critical to high-quality void-free direct wafer bonding.

Surface roughness is crucial to direct wafer bonding as well. It is a microscopic parameter of wafer surface quality, and can be quantified by Root Mean Square (RMS) or surface mean roughness R_a , measured by scanning tunneling microscopy (STM) or atomic force microscopy (AFM). According to Ref [50], direct wafer bonding at room temperature through hydrogen bonds can be realized when R_a is smaller than 0.5nm. With the development of semiconductor technology, Si wafers with roughness smaller than 0.1nm are presently able to be manufactured. In addition, chemical mechanical polishing (CMP) is considered to be an essential technique in direct wafer bonding, which can reduce the surface roughness to an acceptable level for bonding.

Surface flatness is another critical factor for achieving high-quality direct wafer bonding. Surface flatness is defined as the macroscopic deviation of the front surface of a wafer from a specified reference plane, assuming the back surface of the wafer to be flat [50]. The wafer pair needs to overcome the deviation in flatness and deform into a common shape when bonding two well-polished wafers together [75]. The total thickness variation (TTV) is defined as the difference between the highest and lowest elevation of the top surface, which is used to quantify the surface flatness of a wafer. Successful direct wafer bonding can be obtained when bringing two sufficiently-polished wafers into intimate contact at room temperature, if the flatness variations between these two wafers are small enough. Nevertheless, unbonded voids will appear when the variation in flatness becomes too large. When the lateral extension R is much larger than the height of void h , as shown in *Figure 29(a)*, the void caused by flatness nonuniformity can be eliminated under the condition that:

$$h \leq R^2 [\gamma / (1.2Ed^3)]^{1/2} \quad (8)$$

where d is the thickness of one wafer. The void is much easier to be eliminated for thinner wafers, when the lateral extension is similar to the height of the void and is

much smaller than the thickness of wafer d . For heights smaller than the critical height as illustrated in *Figure 29(b)*, the void can still be eliminated if:

$$h \leq 2.6(R\gamma / E)^{1/2} \quad (9)$$

The critical void height is defined as:

$$h_{crit} \leq 3.7(d\gamma / E)^{1/2} \quad (10)$$

Figure 29. (a) Schematic of voids between wafers when $R > 2d$ and (b) when $R \ll 2d$ [49].

If the heights of all voids fall below h_{crit} , then void-free bonding can be easily realized without any macroscopic unbonded areas. It has been reported that flatness variations of between 1 to 3 μm can be accommodated via elastomechanical deformation; a wafer bow of up to 25 μm can be tolerated in direct wafer bonding.

The voids at the bonding interface can be divided into two kinds: one caused by the aforementioned extrinsic particles, which can be eliminated by pre-bonding cleaning or proper wafer handling; or the use of high-level cleanroom environments, of Class 10 and above. The other example is intrinsic voids, also known as thermal bubbles. Thermal bubbles originate at the bonding interface from the by-products of the chemical reaction and will severely degrade bonding quality. In hydrophilic bonding, the bonding is first initiated between two mating surfaces via Van Der Waals forces or hydrogen bridge bonds. Generally speaking, hydrogen bonding can happen at room temperature between one material that can form H-F, H-O, or H-N bonds, or that can attach electrophilic hydrogen on its surface with another of the same or a different material, together with a surface containing a sufficient density of nonbonding electrons of oxygen, nitrogen or fluorine. In addition to the basic requirements for

direct wafer bonding (a clean, flat, smooth, and particle-free surface), activation treatments are also required to obtain hydrogen bonding if the bonding surfaces are covered with silicon oxide or nitride. Treatment with chemicals such as OH⁻, H⁺, or F⁻ will attack the Si-O-Si or Si-N-Si bond to form Si-OH, Si-F, or Si-NH groups; whereas treatment with plasma can introduce bond defects to enhance chemical reactivity [76]. Thus, bonding can be strengthened by post-bonding annealing, with the release of bonding by-products such as H₂O and H₂. In Si-OH group-based bonding, the chemical reaction that happens during post-bonding annealing can be described as follows:

This polymerization reaction between Si-OH groups can occur at temperatures around 100°C. During the post-bonding annealing process, very few bonding by-products can laterally diffuse out of the bonding interface from the edge of the bonding pair. Since the bonding strength is strong, most bonding by-products will be trapped at the interface [49, 77]. Then, the trapped by-products, mainly H₂O, will diffuse through the oxide or nitride layer and oxidize the silicon substrate at temperatures between 200 and 800°C [78, 79]. The oxidation reaction can be described as:

The release of hydrogen will cause thermal bubbles to bulge up. To eliminate these bubbles, the bonded wafer has to undergo high temperature annealing at above 900°C [78]. However, high temperature treatment carried out at 900°C and above may cause undesired dopant diffusion or degradation of the bonding structure. Furthermore, high temperature treatment is entirely incompatible with the Back-End-Of-Line (BEOL) process, which is believed to be the most suitable process to realize integration between electronics and photonics. Therefore, it is critical to find other possible methods to solve the problem of bulging thermal bubbles. Mack *et al.* reported that gases in the thermal bubbles comprise mainly CH₄, from hydrocarbon decomposition, and H₂ in both hydrophilic and hydrophobic bonding [80]. The content in the thermal bubbles caused by hydrocarbon is negligible, leaving the main species in the thermal bubbles to be H₂. H₂ can actually dissolve in the oxide; therefore, a thick oxide layer in the

bonding structure can help to suppress thermal bubbles bulging up. It is suggested that the critical thickness of the silicon oxide layer, in order to fully dissolve H_2 , is approximately 30nm [81]. In the GON wafer fabrication process, a thicker oxide layer is applied to guarantee a high-quality void-free wafer bonding.

3.3 Experimental Preparation Procedures

3.3.1 Ge Epitaxy Deposition and Characterization

Before the GON wafer bonding and layer transfer processes, a high-quality Ge layer with low threading dislocation density (TDD) was first deposited on a Si donor substrate. An 8-inch Si (001) donor wafer was first cleaned using the RCA cleaning method, as described in section 3.2, and then loaded into a reduced pressure chemical vapor deposition (RPCVD) reactor—the ASM Epsilon 2000 Reactor. The three-step Ge growth method was used to obtain a high-quality Ge layer with a thickness of 1.5 μm [82]. Firstly, the low temperature growth at 400°C ensured a very smooth and continuous Ge seed layer. Secondly, the temperature was increased from 400°C to 600°C at a rate of 6.5°C/min, where the flow of the Ge precursor was reduced to maintain reactor integrity and continue with Ge film growth at a lower growth rate. The purpose of the slow ramp rate was to allocate sufficient time for the thermal exposure of the underlying Ge layer, to reduce threading dislocation while maintaining Ge growth. Finally, the temperature was maintained at 600°C to increase the deposition rate, controlling the overall deposition time. After the deposition of the Ge layer, the wafer underwent post-deposition thermal cycling for 8 times in hydrogen ambient at a higher temperature than the growth temperature, ranging from 680 to 825°C, to enhance the surface mobility of Ge atoms. This resulted in a reduction of the final surface roughness, with a RMS of 0.9nm, measured by AFM as shown in *Figure 30* below. The TDD level of the Ge layer after post-deposition annealing was estimated by counting etch pits after iodine etching (a mixture of $HF/HNO_3/CH_3COOH=5:10:11$) as the threading dislocations can be etched faster than the film in the iodine etch solution [83]. The etching pits are visibly spotted under a Nomarski microscope and SEM; thus, TDD can be estimated from the number of etching pits counted in the specified area,

which is $\sim 6 \times 10^6 \text{ cm}^{-2}$ in this case. High-resolution x-ray diffraction (HRXRD) was used to determine the quality and strain of the Ge epitaxial film, since the strain of the final Ge epilayer has considerable influence on its electrical and optical properties.

Figure 30. (a) AFM measurement results showing a RMS roughness of 0.9nm and (b) a cross-sectional SEM image of 1.5 μm Ge deposited on Si substrate.

Figure 31. High-resolution x-ray diffraction (HRXRD) profile showing the crystallinity and strain state of the Ge epilayer in GOS after film deposition and post-deposition annealing.

The HRXRD result in *Figure 31* shows that the Ge epilayer has a high crystal quality. The Ge peak signal is asymmetric and it is broadened at the sides towards higher incidence angles, resembling a “shoulder”. This broadening shoulder can be attributed to Ge/Si intermixing at the interface during thermal processing, perturbing the abrupt interface as a result of Si diffusion into the Ge epilayer to form an intermediate SiGe layer. The position of the Ge peak is shifted to the right, with reference to the Ge bulk substrate, as a result of tensile strain. This tensile strain can be calculated according to Bragg’s law. The perpendicular lattice constant $a_{Ge\perp}$ can be calculated from the HRXRD diffraction peak:

$$a_{Ge\perp} = \frac{2\lambda}{\sin(\frac{\omega_{Ge}}{2})} \quad (13)$$

where λ is the incident radiation wavelength (Cu $K\alpha_1$ line, $\lambda=1.5406$ Å) and ω_{Ge} is the angular position of the Ge peak from the HRXRD, which was measured to be 66.0595° , and $a_{Ge\perp}$ which was estimated to be 5.6529 Å, based on equation (13). Therefore, the in-plane lattice constant $a_{Ge\parallel}$ can be calculated with the equation below:

$$a_{Ge\parallel} = (\frac{1+\nu}{2\nu})[a_{Ge} - a_{Ge\perp}(\frac{1-\nu}{1+\nu})] \quad (14)$$

where ν is the Poisson’s ratio of Ge, taken as 0.271. Therefore, the in-plane lattice constant is estimated to be 5.6644 Å, taking the unstrained Ge lattice constant a_{Ge} as 5.6578 Å. The strain of the film can be expressed as:

$$\varepsilon = (a_{Ge\parallel} - a_{Ge}) / a_{Ge} \quad (15)$$

Thus, the tensile strain of the Ge epilayer was calculated to be 0.12%. This is thermally induced in the Ge epilayer during cooling after high temperature growth, as Ge and Si have different linear coefficients of thermal expansion [84]. This small tensile strain residing in the Ge epilayer will not affect its transparency in the Mid-IR range [85].

3.3.2 SiN_x Deposition and Wafer Bow Engineering

Once the high-quality Ge-on-Si wafer was ready, a SiN_x layer could be deposited on it to prepare for the wafer bonding process. Thus, 1 μm SiN_x was deposited on top of the Ge layer, using plasma-enhanced chemical vapor deposition (PECVD) in an AEGIS20 Cello PECVD reactor. The precursors used for SiN_x deposition were SiH₄, NH₃, and N₂, with the gas flow ratio being 1:10:20. The deposition process was carried out under the temperature of 300°C with RF power of 50W and pressure of 400mTorr. The chemical reaction in the reactor can be depicted as below:

However, it is very difficult to produce a perfect stoichiometric SiN_x with $x=4/3$. The actual composition of the films can vary within certain ranges by adjusting the deposition conditions, including reactant gas flow ratio and pressure, substrate temperature, and plasma power [86-88]. Thus, the accurate n,k coefficients of the deposited SiN_x film have to be measured before wafer bonding and device design, as different compositions would lead to different n,k numbers. As shown in (16) and (17), hydrogen and ammonia will be generated during these chemical reactions, and these gases will be trapped within the film and cause thermal bubbles in the subsequent wafer bonding process, as they diffuse to the bonding interface to form thermal bubbles during the later thermal treatment processes. As mentioned in section 3.2, thermal bubbles pose as a severe threat to bonding quality and may cause debonding. Thus, it is critical to release the by-products generated during the deposition and bonding reaction as much as possible. Therefore, a post-deposition annealing process has to be inserted into the wafer bonding process to ensure the release of gases trapped in the film during deposition. After SiN_x deposition, the wafer was loaded into a rapid thermal annealing system to conduct the SiN_x densification process in N₂ ambient at 450°C for 7 hours. After the post-deposition annealing process, the wafer was inspected under a Nomarski microscope and the images are shown in *Figure 32*.

Figure 32. Nomarski microscope images were taken after the annealing process. (a) clear cracks can be seen on the SiN_x film and some parts of the SiN_x peeled off. (b) cracks also can be seen on the Ge layer after removal of the SiN_x film.

As can be seen from *Figure 32(a)*, the SiN_x film cracked and some parts peeled off after post-deposition annealing. These crack lines could still be observed on the Ge layer, even after removal of the SiN_x film. In order to find out the reason behind the microcracks on the Ge layer after SiN_x deposition and post-deposition annealing, the

bow measurement was conducted after each step, with the stress residing in the SiN_x film calculated using the formula described below [89]:

$$\sigma = \frac{Eh^2}{(1-\nu)6Rt} \quad (18)$$

where E is Young's modulus, ν is Poisson's ratio, h is wafer thickness (m), t is film thickness (m), and R is the radius of curvature of wafer (m), which is defined as:

$$\frac{1}{R} = \frac{1}{R_2} - \frac{1}{R_1} \quad (19)$$

where R_1 is the average radius of the substrate before film deposition and R_2 is the radius after deposition, which can be measured using equipment FLX 2320 for bow measurement. According to the measurement results, the stress residing in the SiN_x film after deposition was -6.35×10^6 Pa of compressive stress; after post-deposition annealing, this stress changed from compressive stress to tensile stress, which measured 5.39×10^8 Pa. Thus, the large change in stress before and after post-deposition annealing deformed the wafer drastically, which resulted in the cracks on the thin film. In order to solve this problem, a SiN_x film with the same thickness was deposited on the back of the Si donor wafer under the same condition, to compensate for the large stress change caused by the SiN_x film deposited on the front side of the wafer after post-deposition annealing. The wafer was checked under a Nomarski microscope again, with no cracks found this time, as illustrated in *Figure 33*.

Figure 33. No crack was found after post deposition annealing through depositing SiN_x film on both side of the GOS wafer.

In order to ensure that the SiN_x deposition and post-deposition annealing processes would not affect the bonding process, the bow measurement had to be conducted before and after SiN_x deposition and post-deposition annealing, to make sure that the wafer bow was within the acceptable range for wafer bonding. The measurement results after each step are shown in *Figure 34*.

(Caption on the next page)

Figure 34. (a) the wafer bow of the GOS wafer before SiN_x deposition. (b) the wafer bow after SiN_x films deposited on both sides of the GOS wafer and (c) after the post deposition annealing.

From the wafer bow measurement results, the stress induced by the SiN_x films is almost balanced, with the final wafer bow measuring $16.6 \mu\text{m}$ after deposition of SiN_x on both sides of the wafer and post-deposition annealing, which is smaller than the bonding criterion of $25 \mu\text{m}$. Thus, it is suitable for wafer bonding. Before wafer bonding, the n, k coefficients of the deposited SiN_x film after densification was measured by an ellipsometer, and the result is shown in Figure 35 below. It indicates that the SiN_x film is transparent up to $7.5 \mu\text{m}$ as suggested by Lambert's law of absorption $\alpha = 4\pi k / c$ where α is the absorption coefficient, and k is the extinction coefficient. It has a refractive index of 1.9 at the wavelength of $3.8 \mu\text{m}$.

Figure 35. The measurement result of the n, k coefficients of the deposited SiN_x demonstrating that there is no light attenuation at wavelength smaller than $7.5 \mu\text{m}$.

3.4 Wafer Bonding and Layer Transfer of GON

In order to initiate the bonding process, a bonding layer was required to be deposited on top of the SiN_x film to facilitate bonding. Thus, a 300nm bonding layer of SiO_2 was deposited on top of SiN_x using low temperature PECVD. Another 300nm of SiO_2 was deposited on top of the Si handle wafer. After the deposition of SiO_2 , the bonding layers were subjected to chemical mechanical polishing (CMP) down to 100nm, in order to reduce surface roughness for the layers to be suitable for wafer bonding. As mentioned before, a thick oxide layer is preferable for high-quality bonding as the by-products from the bonding reaction can be dissolved in the oxide layer. According to reference [81], a 30nm oxide layer is thick enough to realize void-free bonding. In this work, oxide bonding layers up to 200nm thick were applied to guarantee bonding quality. Prior to wafer bonding, both wafers were subjected to O_2 plasma exposure for about 15 seconds, followed by rinsing with deionized (DI) water and then spin dried in a spin rinse dryer. O_2 plasma exposure is able to increase the surface hydrophilicity of the SiO_2 bonding layers; and the rinsing step was necessary to clean the wafer surfaces as well as populate the surfaces with hydroxyl groups at a sufficiently high density to initiate wafer bonding.

Since SiO_2 was used as the bonding layer and with the fact that SiO_2 is not transparent in the Mid-IR range beyond $3.8 \mu\text{m}$, the question arises to whether the $1 \mu\text{m}$ SiN_x film is thick enough to prevent light from being absorbed by the SiO_2 bonding layer. A simulation using Lumerical MODE SOLUTION was done to verify this question and the simulation result is shown in *Figure 36*.

Figure 36. The simulation result of the thickness of SiN_x film vs the power leaking in the bonding layer.

In the simulation, unit power was launched into a planar Ge waveguide on the GON wafer at the wavelength of $3.8 \mu\text{m}$, and the power leaking into the bonding layer was plotted as a function of the thickness of the SiN_x film. The plot illustrates that little power leaked into the bonding layer when the thickness of the SiN_x film was $1 \mu\text{m}$. This is mainly because the bonding layer is too thin to absorb light, and that there have been recent reports stating that SiO₂ can support light at the wavelength of $3.8 \mu\text{m}$ due to the large contrast in refractive index with the core material [90]. Thus, $1 \mu\text{m}$ SiN_x is thick enough to confine the light to a wavelength of around $3.8 \mu\text{m}$. The process flow for wafer preparation is described in Figure 37.

Figure 37. The wafer preparation before wafer bonding and layer transfer to realize GON wafer.

To conclude the wafer bonding preparation process, firstly, a high-quality Ge-on-Si wafer was obtained by the “three step growth” method, whose TDD was only at the order of 10^6 and surface roughness was only 0.9nm. Secondly, a $1\ \mu\text{m}$ SiN_x was deposited on top of the Ge layer and post-deposition annealing was done to release the gases trapped in the wafer during the deposition process. The same process was done to deposit another $1\ \mu\text{m}$ of SiN_x on the back of the wafer to balance the stress. Thirdly, a 300nm bonding layer of SiO_2 was deposited on top of the wafer and the Si handle wafer by LTPECVD, respectively. Finally, the wafers were subjected to CMP to reduce the bonding layer to 100nm; then, the wafers were exposed to O_2 plasma to increase its hydrophilicity, which was followed by DI water rinsing. After all of these processes, the wafers were ready for bonding to obtain the GON structure.

The wafer bonding and layer transfer processes are illustrated in *Figure 38* below.

Figure 38. Schematic of wafer bonding and layer transfer processes.

As mentioned above, both the Si donor wafer and Si handle wafer were exposed to O_2 plasma to increase surface hydrophilicity. Then, these two wafers were brought face-to-face to form intimate contact and were then loaded into the wafer bonder SUSS MICROTEC MA/BA6 to initiate the bonding process at room temperature. After bonding, the bonded wafer pair was annealed at 300°C in an atmospheric N_2 ambience for 3 hours to enhance the bond strength as described in (11). The bonded wafer was inspected under IR imaging to check for the formation of interfacial voids before and after the annealing process. IR imaging is a fast and cost-effective method to detect bonding defects. It utilizes the transmission of infrared radiation through a silicon

substrate which is transparent to radiation with wavelengths larger than $1.1\ \mu\text{m}$. The bonding defects are observed as dark spots, voids, or interfacial lines under IR imaging.

Figure 39. IR imaging inspection before and after the annealing process after wafer bonding.

Under IR imaging, no voids and particles were found in the interface after the bonding process. Normally, the annealing process would introduce several voids in the interface because the gas trapped inside will be released during the annealing process. This can be caused by a rough surface of the bonding layers. Our method employed CMP before wafer bonding; whereby it gives a flat and smooth surface that can significantly improve the bonding quality. After bonding, the Si donor wafer was removed by grinding, followed with tetramethylammonium hydroxide (TMAH) wet etching. At the same time, the Si layer from the handle wafer was protected with a ProTEK B3-25

coating. The TMAH has a high etching selectivity on Si over Ge, and stops at the Si/Ge intermixing layer, where misfit dislocations happen. This misfit dislocation can cause high scattering loss for waveguides on the normal GOS platform and is difficult to remove, as it resides at the interface between Si and Ge. Nevertheless, this defective layer can be exposed on top of the surface and removed by CMP through the wafer bonding and layer transfer process. Thus, the Ge layer was thinned down to 1 μm by CMP and the AFM measurement result and the cross-sectional image of the bonded GON wafer are shown in *Figure 40*. The bonded wafer did not debond during the mechanical grinding and CMP process indicating a strong bonding strength.

Figure 40. (a) AFM measurement result showing a RMS roughness of 0.26nm after CMP; (b) Cross-sectional SEM image of the bonded GON wafer.

The final thickness of the Ge layer was about 1 μm , and the thickness of SiN_x was about 0.8 μm . The shiny layer observed in the middle of the silicon nitride layer is a measurement artifact, due to the well-known charging effect of dielectric layers during SEM imaging. The HRXRD was conducted on the Ge layer after wafer bonding and layer transfer to determine the crystal quality and the strain state, with the result illustrated in *Figure 41*.

Figure 41. HRXRD measurement result on the Ge layer of the bonded GON compared to the GOS and bulk Ge.

As can be seen from the measurement result, the peak position of the GON wafer shifted to the left a little, compared to the GOS, but was still located to the right of the bulk Ge, indicating tensile strain. The tensile strain was calculated to be 0.07% and the relaxation of tensile strain was attributed to the removal of the Si donor wafer. The crystal quality of the Ge layer on GON did not change significantly, as indicated by the peak intensity and curve shape, which are comparable to that of the Ge-on-Si wafer. The full width half maximum of the GON curve increases from 176.78 arc sec to 182.52 arc sec, indicating a slight degradation in film quality which can be attributed to improper storage of the sample in a humid environment. Nevertheless, the wafer bonding and layer transfer process for GON fabrication does not have any significant influence on the quality of the Ge layer. Thus, the platform which has a large refractive

index contrast between the core and under-cladding layer ($\Delta n=2.1$ @ $3.8 \mu m$), as well as a wide transparency range up to $7.5 \mu m$ in Mid-IR, was fabricated.

3.5 Summary

In this chapter, the fabrication process of the novel GON platform has been described in detail. Before the wafer bonding and layer transfer process, the bonding criterion was studied for a successful bonding process. The bonding surface is required to be smooth and the RMS of the surface is required to be smaller than 0.5nm for high-quality bonding. These requirements can be satisfied by CMP. The flatness of the wafer is another critical factor for void-free bonding, especially for wafer-level bonding. Thus before bonding, the wafer bow has to be measured and controlled within $25 \mu m$. The reason behind the thermal bubbles has been explored and the solution for this problem found. By using a thick oxide layer as the bonding layer, the by-products, especially H_2 , can be dissolved in this layer, providing void-free bonding. By applying SiN_x as the under-cladding layer, the refractive index contrast between core and clad can become larger, and the transparency window can also be extended to $7.5 \mu m$, compared to normal SOI or GOS. However, the large stress change after post-deposition annealing of the SiN_x film causes cracks on the Ge layer. In order to solve this problem, a SiN_x film with the same thickness was deposited on the back of the wafer to compensate for the stress. Ellipsometer measurements on the SiN_x film suggest that it has a refractive index of 1.9 at the wavelength of $3.8 \mu m$, with its extinction coefficient remaining at zero up to about $7.5 \mu m$. After the wafer bonding and layer transfer process, the bonded wafer was inspected under IR imaging and no defects were found, indicating a high-quality bonding process. The Ge layer was characterized under HRXRD, showing a 0.07% tensile strain attributed to the bulk Ge, with no significant degradation in crystal quality after the bonding process. This result indicates that the wafer bonding and layer transfer technique can be applied to all wafer levels and has no influence on the quality of materials. This technique and all materials involved are entirely CMOS-compatible, which makes it promising for integration between electronics and photonics. The novel GON platform will have a major impact on photonic integrated circuits and Mid-IR

sensing applications, due to its wide transparency range in Mid-IR, as well as its large refractive index contrast which makes compact devices possible.

Chapter 4 Waveguide Design, Fabrication and Characterization on GON

The methods of propagation loss measurement were studied, in order to guide the chromium mask design of waveguides, as well as to decide the components for building up an optical testing platform. The optical testing platform was set up for waveguide characterization and the details will be described in section 4.2. The influence of fabrication conditions on the performance of waveguides was first studied on a GOS platform. Single-mode rib waveguides were fabricated on the GOS platform with different dry etching processes, and the performance of the waveguides presented a large difference, indicating that the fabrication process has a prominent influence on the loss performance of waveguides. By applying proper fabrication conditions, it becomes essential to design low-loss waveguides on the GON platform. The theoretical calculation, modelling, fabrication, and characterization of the single-mode strip waveguide were realized on the GON platform, as well as the low-loss sharp bent waveguide, to demonstrate the advantages of the GON platform. Strip structure is chosen due to the poor mode confinement compared to rib structure thus the evanescent field fraction can be extended resulting in a higher sensitivity.

4.1 Waveguide Loss Characterization Method

The propagation loss and the bending loss measurements can be completed by employing the cut-back method, which is one of the simplest and most accurate methods of measuring waveguide loss [91]. This method requires waveguides with varied length fabricated on the platform to characterize the output power change and then equation (20) is used to calculate propagation loss in dB/cm. Light of the desired wavelength being focused onto a cleaved or polished input face of a waveguide and being received at the output end of the waveguide after passing through it, as schematically demonstrated in *Figure 42*.

Figure 42. Schematic experiment set-up for measurement of waveguide loss using end-fire coupling.

Such direct coupling is referred to as butt-coupling or end-fire coupling [92]. Before each measurement, the laser beam and the sample have to be carefully aligned by maximizing the observed output power. This method is based on a comparison of transmission, through waveguides having a different number of bends or different lengths, as the different lengths or number of bends will attenuate the light differently, resulting accordingly in the drop of transmission at the output end. Thus, the dependence of the transmission on the number of bends or length differences can be plotted, assuming identical coupling conditions and identical surface roughness. The loss coefficient can be determined from the slope of the transmission versus length curve in the following equation:

$$\alpha = 10 \frac{\log_{10}(P_1 / P_2)}{\Delta l} \quad (20)$$

where P_1 and P_2 are the transmitted power for waveguides of two different lengths Δl . According to this method, the waveguides on chromium masks must have different lengths, but are otherwise identical, which means that the number of bends to introduce length differences must be the same for all waveguides. This pattern of the waveguides, commonly termed the paper-clip pattern, is widely used in loss measurement [93]. For the optical testing platform, the equipment required for this method are, simply, a laser light source with the desired wavelengths, fibers to connect the laser source and optical detector to the device under testing, and an optical detector.

4.2 Optical Testing Platform Set-up

In order to explore applications of GON in the Mid-IR range, a laser source with its wavelength tunable from 3.65 to 3.90 μm was equipped in the optical testing platform. It was a rapid-scan CW-Pulsed quantum cascade laser from DAYLIGHT SOLUTIONS with modal number 41038-014-D0108. Its output power under continuous wave mode (CW), as a function of current and wavenumber, is shown in *Figure 43*. As can be seen from *Figure 43*, the laser is triggered at current around 530 mA and reaches its highest output power at wavelength around 3.76 μm .

Figure 43. The data sheet of the Mid-IR laser showing the output power as a function of current and wavenumber.

The real output power of the laser at the current of 725 mA was measured by directly guiding the light from the laser, using a single-mode ZrF₄ fiber from Thorlabs, to a 919P thermopile sensor from Newport at the output end of the fiber. The power was plotted as a function of the wavelength scanning from 3.66 to 3.86 μm at a step of 2 nm and the result is shown in *Figure 44*.

Figure 44. The actual power of the laser measured at the current of 725 mA as a function of the wavelength.

As can be seen from the data sheet and the actual measurement result, there is a large difference in the shape of the curve, as the power fluctuates drastically in the actual situation. In addition, the power is smaller than that of on the data sheet, due to the coupling loss from the fiber tip to the sensor. Nevertheless, the power at the desired wavelength is still large enough to measure waveguide loss and can be used for Mid-IR sensing.

The optical detector at the output side is an InSb photodiode photodetector from HORIBA, whose spectral range covers 1.0 to 5.5 μm and whose sensitivity measures about -40 dB. It converts the power from waveguides into voltage and shows the reading on the display. It has two responsivities, 10^5 and 10^4 V/W, which can be switched by the lever on the photodetector. Since voltage readings on the photodetector have a linear relationship with the power coming from the waveguides, the propagation loss can be directly calculated, using the readings from the photodetector, as:

$$\alpha = 10 \frac{\log_{10}(V_1 / V_2)}{\Delta l} \quad (21)$$

The ZrF_4 fiber from Thorlabs was used to couple the light from the laser source into the devices under testing, as well as collect the light passing through waveguides to the photodetector. It has a wide transmission range from 285 nm to 4.5 μm , but for single

mode operation, the wavelength ranges from 2.3 to 4.1 μm . The core diameter measures 9 μm and its attenuation for wavelength, from 2.3 to 3.6 μm , is smaller than 0.2 dB/m, therefore it can be ignored. In order to reduce background noise and improve measurement accuracy, a lock-in amplifier was employed in this system. A lock-in amplifier is a type of amplifier that can extract a signal with a known carrier wave from an extremely noisy environment. Since the sensitivity of the InSb photodiode optical detector is only about -40 dB, which is only half of what commercial detectors usually use at 1.55 μm , it is necessary to integrate a lock-in amplifier in the system to reduce noise, as well as amplify the signal coming from the optical detector. To add the carrier wave, a chopper was installed in front of the laser source to add a carrier wave with the frequency of 267 Hz. The lock-in amplifier was set to be only sensitive to this frequency, thus blocking other frequencies from the background. A polarization controller was added in front of the chopper to adjust the polarization of the light. Following the polarization controller, the light is focused into the fiber by a lens. The set-up is shown in *Figure 45*, and the schematic of the entire testing system is illustrated in *Figure 46*.

Figure 45. Chopper and polarization controller added in front of the laser to provide carrier wave as well as control the polarization of the light.

Figure 46. Schematic of the optical testing platform.

It is impossible to operate so many instruments manually at the same time, therefore using Labview to control the instruments through a computer is crucial for fast and effective waveguide loss measurement. The block diagrams and operation interfaces for the laser, photodetector, and lock-in amplifier are shown in Figure 47.

(b)

Figure 47. (a) the block diagram of connecting lock-in amplifier, chopper, and photodetector. (b) the operation interface of the lock-in amplifier. (c) the block diagram of connecting laser, chopper, lock-in amplifier, and photodetector. (d) the main operation interface for waveguide loss measurement.

Before running the laser, the chopper is activated by pressing the “Run Chopper” button on the operation interface. Then, the current value for the laser needs to be typed in, which is normally 680 mA. This current can provide enough output power for measuring waveguide loss; moreover, a lower current is ideal for preserving the service life of the laser. Finally, the target wavelength is typed in, followed by clicking the start button to run the program. So far, both the software and hardware for the optical testing platform have been accounted for, from which waveguides can be fabricated and characterized.

4.3 Waveguide Fabrication and Optimization of the Fabrication Process

The fabrication processes of waveguides are standard processes identical to CMOS foundry industries, including cleaning, photoresist coating, optical lithography, developing, etching, and photoresist stripping. These processes will have a critical influence on the performance of waveguides, especially regarding loss, as particles in clean rooms as well as surface roughness after dry etching would cause scattering loss, thus increasing the total loss of the waveguide. Normally, losses in waveguides

originate from several sources, a notable one being absorption due to metal wires and contacts. In order to avoid this kind of absorption, proper design and the distance between the metal and waveguides need to be considered. The main source of loss is scattering loss, caused by sidewall roughness and contaminants on the side walls of waveguides. A modal for the theoretical analysis of scattering loss was proposed by Payne *et al.* and the schematic of the modal is shown in Figure 48 below [94].

Figure 48. The schematic of the modal for scattering loss estimation [91].

The estimation of the scattering loss in planar optical waveguides, due to irregularities in the waveguide walls, can be expressed as:

$$\alpha \leq \frac{\sigma^2}{k_0 d^4 n_1} \kappa \quad (22)$$

where α is loss in m^{-1} , k_0 is the free space wavenumber, n_1 is the refractive index of waveguide, d is half the width of the waveguide, and σ is the sidewall roughness in RMS. κ corresponds to 0.48 for exponential statistics, and 0.76 for Gaussian. After dry etching to form a vertical sidewall for the waveguide, the sidewall roughness can be measured by AFM; thus the scattering loss from sidewall roughness can be estimated [95]. According to equation (22), it is important to reduce sidewall roughness to decrease the scattering loss of the waveguide. An advanced fabrication process can reduce sidewall roughness, by improving the lithography process with a cleaner exposure environment and a better exposure source, like electron beams, to provide a clean surface and a photoresisting vertical sidewall after developing. A better dry etching recipe with different etchants may also give a more vertical etching profile and smoother sidewalls [96]. The influence of the fabrication process on the performance of waveguides will be discussed later on in this chapter.

Besides sidewall scattering loss, material loss is another factor attributed to waveguide loss due to dopants absorption, untransparency of semiconductors within certain wavelength ranges, and resonance between light frequency and vibration of molecules. For passive devices on undoped GON platforms in Mid-IR range, the material loss is negligible. Surface-state absorption can also contribute to propagation loss if the waveguides are not properly passivated. Since the main contribution to propagation loss is scattering loss, care must be taken during the fabrication process to reduce particle contaminants as much as possible. Optimizing the fabrication process is also essential to produce low-loss waveguides, by improving lithography technology and dry etching process to promote the etching profile, as well as to reduce sidewall roughness. The fabrication techniques and optimization process was first conducted on a 6-inch Ge-on-Si wafer with Ge thickness of $3\ \mu\text{m}$. The design was from a collaborator which was a single-mode rib waveguide, with a width of $2.7\ \mu\text{m}$ and etch depth of $1.5\ \mu\text{m}$ for the operation wavelength of $3.8\ \mu\text{m}$. The light was coupled into the waveguides by a grating coupler, whose grating pitch was $2\ \mu\text{m}$ with the duty cycle of 0.5. Thus, the critical size of this layout was $1\ \mu\text{m}$, which can be realized by a wide range of lithographies. The fabrication process is described as follows.

Firstly, the GOS wafer was cleaned by immersing it in Aceton, IPA, and DI water for 5 minutes, respectively, to remove organic contaminants and particles. After that, the wafer was blow-dried by an N_2 gun and baked on a hot plate at 105°C for 4 minutes to completely remove moisture on the wafer. Then, the wafer was coated with positive photoresist AZ 5214, using a spin coating machine at the spin speed of 4000 r/s for 30s, with the thickness of the photoresist being about $1.4\ \mu\text{m}$. After coating, the wafer was baked at 105°C for 2 mins, which is commonly known as the soft bake process. Then, the wafer was ready for the exposure process.

Figure 49. Schematic of the sample after photoresist coating.

Secondly, a 365nm *i*-line MJB4 Mask Aligner was used to conduct the optical lithography process. After the mask and the sample had been contacted, the exposure process was conducted. Following exposure, the developing process was conducted by immersing the sample into CD26 developer for approximately 20 seconds, until the exposed photoresist completely peeled off from the surface of the wafer. Then the sample was rinsed by DI water immediately for 30 seconds to totally wash the developer away.

Figure 50. Schematic of the sample after optical lithography

Thirdly, the wafer was loaded into a RIE Oxford Plasmalab 80 for the dry etching process, with Cl_2 gas as the etchant to conduct reactive ion etching (RIE). The power of etching process was 200 W, which was the maximum power of the machine. The chamber pressure was set to be 20mTorr, with the gas flow rate being 50 sccm. The etching rate was about 4.6nm/s. The etching selectivity between Ge and the photoresist is 7:1.

Figure 51. Schematic of the sample after etching process.

After etching, the photoresist residue was completely removed by O_2 plasma, such that it would not affect the measurement results of propagation loss and bending loss. The fabricated waveguides and grating coupler were inspected using SEM, and the images are shown in *Figure 52*.

Figure 52. (a) Cross-sectional SEM of grating coupler. (b) sidewall slope observed after RIE.

The mechanism of RIE can be found in Ref [97]. Generally, plasma produced and sustained in a discharge between two electrodes in a RIE chamber consist of charged species, including positive and negative ions as well as electrons, radicals, and neutrals. The radicals and neutrals participate in chemical reactions on the substrate to form volatile species, while positive ions are accelerated by the bias added on the electrodes and bombard wafer surfaces to initiate or complete the volatilization process and accelerate material removal. It can be seen from *Figure 52(b)* that the sidewall was not vertical but presented with an undercut, V . It has been reported by Deri *et al.* [98] that the scattering loss in waveguides is affected by the slope of the sidewall. A vertical sidewall shape results in a minimal scattering loss factor. Anisotropy (A) is here defined as:

$$A = (1 - \frac{V}{H}) \quad (23)$$

The etch depth is defined as H , and the maximal undercut V . According to the SEM image, V was observed to be $0.5 \mu\text{m}$ for the RIE etching profile. A perfect anisotropy etching is represented by $A=1$ but a 0.66 anisotropy was obtained for the RIE-etched waveguide, resulting in a higher loss. This RIE-etched sample was tested on the optical testing platform. Since the light was coupled into the waveguide by the grating coupler with an incident angle of 10° , two special holders for fibers were mounted on the XYZ translation stage, as shown in *Figure 53* below.

Figure 53. The stage for waveguide loss measurement with grating coupler.

Propagation loss of the waveguides fabricated with RIE was measured using the cut-back method and the result is shown in *Figure 54*. The loss was $6.85 \pm 0.5 \text{ dB/cm}$.

Figure 54. The propagation loss measurement result of the waveguides fabricated with RIE.

Waveguides with the same design were also fabricated by the same fabrication process, except using Deep-RIE to etch the waveguides. In the Deep-RIE recipe, the etchant is a mixture of SF_6 and C_4F_8 , with a gas flow of 23 and 65 sccm respectively. The power was at 900 W, and the pressure at 120 mTorr. With different etchants and a much higher power, a sharp and vertical etching profile can be observed by SEM, as shown in Figure 55.

Figure 55. Cross-sectional SEM image of grating coupler fabricated with Deep-RIE.

Compared to the sidewall of waveguides etched by RIE, the sidewall of the Deep-RIE etched profile is nearly vertical ($A=0.933$). Consequently, the scattering loss was reduced to 2.65 ± 0.1 dB/cm, as shown in *Figure 56*. Therefore, a better dry etching process and recipe can provide a vertical sidewall, minimizing propagation loss due to scattering effects. The Deep-RIE process has become a standard process for future waveguide fabrication, and the recipes of RIE and Deep-RIE are listed in Table 3.

Figure 56. The propagation loss measurement result of waveguides etched by Deep-RIE.

Dry Etching Recipe	RIE	Deep-RIE
Etchant	Cl ₂	SF ₆ /C ₄ F ₈
Gas Flow Rate (sccm)	50	23/65
Power (W)	200	900
Pressure (mTorr)	20	120
Etch Rate (nm/s)	4.6	14.6

Table 3. The comparison of recipes between RIE and Deep-RIE.

4.4 Strip and Ultra-compact Bent Waveguides Design and Modelling on GON

When light is coupled into a confined space, different modes will be activated, e.g. radiation mode and guiding mode. The guiding mode means that light is well-confined in media, such as in fiber or planar waveguides, by total internal reflection for transmission from one point to another point. According to the polarization of guided light, modes can be divided into transverse electric (TE) modes and transverse

magnetic (TM) modes. A different number of modes of light can be sustained in waveguides, depending on the size of waveguides as well as their boundary conditions. Normally, single-mode operation is preferred, as multimodal transmission in waveguides may cause higher propagation loss and dispersion of signal [99, 100]. In order to design a single-mode strip waveguide on the novel GON platform, ray theory was employed to theoretically estimate the thickness of the Ge layer first, followed by simulations to decide the width of waveguide for single-mode operation. A bent waveguide was also modelled in simulations, and the results demonstrated that the GON platform can support an ultra-compact bent waveguide with a low bending loss, compared to the normal GOS platform.

Figure 57. The schematic of total internal reflection of light in a three-layer planar waveguide.

According to Snell's law, total internal reflection will happen when

$$\arcsin \frac{n_2}{n_1} < \theta_1 \quad (24)$$

assuming $n_1 > n_2 > n_3$. Equation (24) can be rewritten as:

$$n_2 < N = n_1 \sin \theta_1 < n_1 \quad (25)$$

where N is defined as the effective refractive index. When (25) is multiplied by k_0 , where k_0 is the free space wavenumber, the condition for total internal reflection can be expressed as:

$$k_0 n_2 < \beta = k_0 N < k_0 n_1 \quad (26)$$

where β is defined as the propagation constant. Equation (26) above describes the condition for the guiding mode. As mentioned before, not only will the guiding mode be activated in the waveguide, but other modes can also exist in waveguides. If

$\beta < k_0 n_3$, light will not be totally reflected on both the upper and lower boundaries, which leads to the energy of light radiating to the upper- and under-claddings, causing a high loss. This mode is called the space radiation mode. When $k_0 n_3 < \beta < k_0 n_2$, light will be totally reflected on the upper boundary but also leaks to the substrate, which is the substrate leaking mode. When light is reflected on the interfaces of the upper- and under-cladding layers, a phase shift of the light will be introduced, and this phase shift can be expressed using Fresnel's formula. For TE mode, the total reflection coefficient can be written as:

$$2\phi_{12} = 2 \arctan \frac{(n_1^2 \sin^2 \theta_1 - n_2^2)^{1/2}}{n_1 \cos \theta_1} \quad 2\phi_{12} = 2 \arctan \frac{(n_1^2 \sin^2 \theta_1 - n_2^2)^{1/2}}{n_1 \cos \theta_1} \quad (27)$$

$$2\phi_{13} = 2 \arctan \frac{(n_1^2 \sin^2 \theta_1 - n_3^2)^{1/2}}{n_1 \cos \theta_1} \quad (28)$$

For TM mode, the total reflection coefficients on the two interfaces are:

$$2\phi_{12} = 2 \arctan \frac{n_1^2}{n_2^2} \frac{(n_1^2 \sin^2 \theta_1 - n_2^2)^{1/2}}{n_1 \cos \theta_1} \quad (29)$$

$$2\phi_{13} = 2 \arctan \frac{n_1^2}{n_3^2} \frac{(n_1^2 \sin^2 \theta_1 - n_3^2)^{1/2}}{n_1 \cos \theta_1} \quad (30)$$

However, not all propagation constants that satisfy equation (26) can form the guiding mode, as the guiding mode has to also meet the requirement of constructive resonance at the equiphase surface, which means that the phase difference of two parallel rays at the equiphase surface has to be an integral multiple of 2π . The schematic of two parallel rays transmitting in a three-layer planar waveguide is shown in *Figure 58*.

Figure 58. The schematic of optical path difference between two equiphase surfaces.

According to Figure 58, the optical path difference between the equiphase surface AC and BD are $(AB-ED)$, so the phase shift at B point would be:

$$k_0 n_1 (AB - ED) - 2\phi_{12} - 2\phi_{13} = 2m\pi \quad (m=0,1,2,\dots) \quad (31)$$

Using the geometrical relationship, $(AB-ED)$ can be represented as $2b\cos\theta_1$, hence equation (31) can be expressed as:

$$2bk_0 n_1 \cos\theta_1 = 2m\pi + 2\phi_{12} + 2\phi_{13} \quad (32)$$

Then, equations (27), (28) or (29), (30) are substituted into equation (32), giving the characteristic equation of the asymmetric three-layer planar waveguide as:

$$\gamma_1 b = m\pi + \arctan T_2 + \arctan T_3 \quad (33)$$

$$\gamma_1 = (k_0^2 n_1^2 - \beta^2)^{1/2}$$

$$\gamma_2 = (\beta^2 - k_0^2 n_2^2)^{1/2}$$

$$\gamma_3 = (\beta^2 - k_0^2 n_3^2)^{1/2}$$

$$T_2 = \left(\frac{n_1^2}{n_2^2}\right)^s \frac{\gamma_2}{\gamma_1}, \quad T_3 = \left(\frac{n_1^2}{n_3^2}\right)^s \frac{\gamma_3}{\gamma_1}$$

where m is the order of mode, b is the thickness of core material, and for TE mode $s=0$, while for TM mode $s=1$. The relationship between thickness of core material and number of modes is now given by equation (33).

Thus, the thickness of the Ge layer on the GON platform for single-mode operation can be determined by substituting m with 0 and 1. $m=0$ means that there is only the fundamental mode supported in the waveguide, while $m=1$ indicates that the first-order mode is activated. For the single-mode waveguide on GON, the thickness b should be smaller than the thickness at which first-order mode is supported. Hence, another equation can be derived from equation (33), as such:

$$\frac{\lambda_0}{2\pi(n_1^2 - n_2^2)^{1/2}} \arctan\left[\left(\frac{n_1^2}{n_3^2}\right)^s \frac{(n_2^2 - n_3^2)^{1/2}}{(n_1^2 - n_2^2)^{1/2}}\right] < b < \frac{\lambda_0}{2(n_1^2 - n_2^2)^{1/2}} \left\{1 + \frac{1}{\pi} \arctan\left[\left(\frac{n_1^2}{n_3^2}\right)^s \frac{(n_2^2 - n_3^2)^{1/2}}{(n_1^2 - n_2^2)^{1/2}}\right]\right\} \quad (34)$$

Therefore, the highest thickness of the Ge layer on the GON platform was calculated to be $1.21 \mu\text{m}$ for the fundamental TE mode operation, which means the GON wafer described in Chapter 3, with a $1 \mu\text{m}$ thickness of Ge, can be used for single-mode waveguide design.

The width of the single TE mode waveguide on the GON platform was decided by a simulation software—the Lumerical Finite Difference Time Domain (FDTD) method. As a comparison, single TE mode waveguides on the normal GOS platform were also simulated and fabricated. The model built using FDTD is illustrated in *Figure 59*. The height in the y -direction of the strip waveguide was fixed at $1 \mu\text{m}$, to denote the thickness of the Ge layer. The length in the z -direction was set to be much larger than the width and height values of the waveguide. Then, the effective indices of the fundamental and first-order TE and TM modes were plotted as a function of waveguide width at the input wavelength of $3.8 \mu\text{m}$. Since the waveguide values for fundamental TE and TM mode were required, there was no need to add other higher-order modes in the simulation, as it could cost excessive time to run the simulation; the cut-off condition for the first-order mode was sufficient to make a judgment. The simulation result is shown in *Figure 60*.

Figure 59. The 3D modal of strip waveguide on GON platform.

Figure 60. The simulation result of effective indices of fundamental TE_0 and TM_0 , and first-order TE_1 and TM_1 .

When the effective indices of modes are smaller than the refractive index of Si ($n=3.42$) at the wavelength of $3.8 \mu m$, it means that the modes leaked into the Si substrate and

cannot be supported in the core layer any further. As can be observed from *Figure 60*, when the width of the waveguide was larger than $3.9\ \mu\text{m}$, the first-order mode was activated in the waveguide; thus the width for single-mode operation should be smaller than $3.9\ \mu\text{m}$. Correspondingly, for single TE mode transmission, the width should be smaller than $3\ \mu\text{m}$. The final cross-sectional size of the strip waveguide was chosen to have $2\ \mu\text{m}$ width and $1\ \mu\text{m}$ height. A waveguide of the same size was also simulated on the GOS platform and the mode profiles of waveguides on the two different platforms are shown in *Figure 61*.

Figure 61. (a) mode profile of single TE mode waveguide on GOS. (b) mode profile of single TE mode waveguide on GON.

The cross-sectional sizes of waveguides on both GON and GOS platforms were the same, with $2\ \mu\text{m}$ width and $1\ \mu\text{m}$ height. It is obvious that the GON platform had a

better confinement of mode at the interface between the core Ge and under-cladding layer, as shown in *Figure 61*. There is a large lattice mismatch (4.2%) between Ge and Si; thus, when Ge was deposited directly on the Si substrate, misfit dislocations would be formed along the Ge/Si interface. The poor confinement of mode at the interface makes light sensitive to the misfit dislocations, causing higher scattering loss. Soref *et al.* mentioned in their paper that extra loss would arise from scattering at the Ge/Si interface as well as from mode-tail loss in the Si substrate, both of which would probably increase propagation loss from less than 1 dB/cm to approximately 2 dB/cm [15].

In order to verify the novelty that the large core/clad index contrast of the GON platform will lead to a better confinement of mode propagating in a bent waveguide, simulations were conducted, and bent waveguides on GOS were also simulated as the benchmark. The model, mode profiles, and simulation results are shown in *Figure 62*.

Figure 62. (a) The 3D modal of 90° bent waveguide. (b) the simulation result of transmission as a function of radius of bent waveguide. (c) mode profile of bent waveguide on GON and, (d) mode profile of bent waveguide on GOS platform.

Unit power was launched into a 90° bend, whose inner radius R was varied from 2 to 18 μm , while keeping the cross-sectional size of waveguides on both GON and GOS the same ($2 \times 1 \mu\text{m}^2$). As can be seen from the simulation result, the transmission of

bent waveguides on GON at the output end is nearly equal to 1, regardless of radius size, indicating a low radiation loss on GON. The performance gap between GON and GOS becomes more apparent as the radius decreases, and the bending loss of bent waveguides on GON was 0.03 dB/bend at the radius of $5 \mu m$; while for GOS, the loss was 1.49 dB/bend. This means the performance of GON is about 50 times better than that of GOS. Light mode tends to leak outwards when passing through the bend, causing radiation loss. A simple model was proposed by Marcatili *et al.* [101] to theoretically estimate the radiation loss in bends, which is expressed as:

$$\alpha = \kappa \exp(-cR), c = \beta(2\Delta n_{eff} / n_{eff})^{3/2} \quad (35)$$

where κ is a constant related to the refractive indices of cladding, core materials and waveguide thickness, β the propagation constant, and Δn_{eff} the difference between the effective index n_{eff} and the cladding index. According to equation (35), the smaller the radius is, the larger the overall loss. A large refractive index contrast within the core/clad composite can also contribute to loss reduction, as the mode can gain better confinement, as shown in *Figure 62(c)*. The mode in *Figure 62(c)* was well-sustained on the GON platform, while the mode on GOS moved outwards and leaked to the substrate, indicating an apparent larger radiation loss, as shown in *Figure 62(d)*. As suggested by equation (35), a larger Δn_{eff} can better confine the mode when it passes through the bend. The cross-sectional size of waveguides on GOS was increased to $3 \times 2 \mu m^2$, under the condition that only the single TE mode is supported by the waveguides. The transmissions at the output end of bent waveguides were plotted as a function of radius size, as shown in *Figure 63*.

Figure 63. The simulation result of bent waveguides with different cross-sectional size on GOS compared to that on GON platform.

The simulation result indicates that a larger cross-sectional size can result in a smaller radiation loss in a bent waveguide, but the refractive index contrast remains the dominant factor in radiation loss, as the performance of GON is still superior to that of GOS, having the largest size. The large refractive index contrast of GON not only allows a compact bent waveguide with a small radius, but also a smaller cross-sectional size.

Thus, the simulation results demonstrate that the GON platform can realize an ultra-compact bent waveguide with low loss, compared to the GOS platform which has been studied extensively in Mid-IR photonics.

4.5 Strip and Ultra-compact Bent Waveguide Fabrication and Characterization on GON

After the demonstration of feasibility of the GON platform for realizing ultra-compact bent waveguides through simulation, the waveguides were designed on a chromium mask and then transferred onto the GON platform for characterization. Paper clip

waveguides were designed for propagation loss measurement with the cut-back method; while bent waveguides were also designed, by varying the number of bends. The masks and fabricated waveguides are shown in *Figure 64*.

Figure 64. (a) mask design for bending loss measurement. (b) paper clip mask design for propagation loss measurement. (c) SEM image of fabricated bent waveguide.

Waveguides were fabricated using the standard process described in section 4.3 and etched using the RIE recipe. The SEM images of waveguides on the GON platform are illustrated in *Figure 65*.

Figure 65. SEM images of (a) bent waveguides; (b) straight waveguide; and (c) cross sectional image of strip waveguide on GON.

The propagation loss and bending loss measurements were carried out by employing the cut-back method. Waveguides of total length ranging from 2mm to 22mm, with a step of 2mm, were fabricated. Each waveguide had 8 bends with a radius of $50\ \mu\text{m}$, whose radiation loss can be ignored, according to the simulation result. The device under testing was mounted on a XYZ translational stage. A light source, with $3.8\ \mu\text{m}$ wavelength from a rapid-scan CW-Pulsed quantum cascade laser from DAYLIGHT SOLUTIONS, went through a polarization controller and then was coupled into a single mode ZrF_4 fiber from Thorlabs. Then, the fiber tip was aligned with the facet of

the strip waveguide for coupling the light into the waveguide. For efficient and reproducible coupling from the fiber to single mode waveguides, taper geometry was designed on each side of the waveguides with a total length of 100 μm , long base of 9 μm , and short base of 2 μm . After passing through the device, the light was collected by a similar fiber as that at the input. The coupling loss of the facet was about 3 dB/facet, and output power was analyzed by an InSb photodiode photodetector from HORIBA. Waveguides were also fabricated on the GOS wafer using the same conditions, for the purposes of comparison. The propagation loss measurement results are shown in *Figure 66*.

Figure 66. Measurement results of propagation losses of waveguides on GON and GOS wafers. GON wafer has a propagation loss of $3.35 \pm 0.5 \text{ dB/cm}$, while GOS has a propagation loss of $8.18 \pm 0.6 \text{ dB/cm}$.

It can be seen that the propagation loss of waveguides on GOS is $8.18 \pm 0.6 \text{ dB/cm}$, which is much higher than that of waveguides on GON, whose propagation loss is $3.35 \pm 0.5 \text{ dB/cm}$. The higher propagation loss of the GOS wafer is mainly caused by poor confinement of the propagating mode. This mode has larger overlaps with the sidewall and the interface between Ge and Si, and misfit dislocations exist in the interface because of the lattice mismatch between Ge and Si. However, the misfit dislocations which were previously hidden along the Ge/Si interface can now be exposed on top of the wafer with the layer transfer method, and can be removed by CMP. In fact, the defective layer was removed, when CMP was performed to thin down the Ge layer to 1 μm after wafer bonding and layer transfer. The propagation loss

of GON can be further reduced, with a better quality of the sidewalls of waveguides to reduce scattering loss, and high-quality sidewalls can be realized by using advanced E-beam lithography and Deep-RIE. Bent waveguides with a radius of $5\ \mu\text{m}$ were also fabricated on GON. For comparison, bent waveguides with radii of 5, 15 and $20\ \mu\text{m}$ were fabricated on the GOS wafer. The number of 90-degree bends on both wafers varied from 4 to 36, and the bending loss measurement results are illustrated in Figure 67.

Figure 67. Measurement results of bending losses on GON and GOS.

As shown in the plot, the bending loss of waveguides on GON is much smaller than that on GOS due to better confinement, and its bending loss is only $0.14 \pm 0.01\ \text{dB}/\text{bend}$, compared to $2.54 \pm 0.15\ \text{dB}/\text{bend}$ on GOS at the radius of $5\ \mu\text{m}$. Even when the radius of bent waveguide was increased to $20\ \mu\text{m}$, the bending loss on GON platform was still smaller than that on GOS. The influence of the change in length was ruled out when calculating bend loss using the propagation loss of $3.35\ \text{dB}/\text{cm}$. The higher bending

loss of GOS is due to the mode interacting intensively with the interface and sidewall, due to poor optical confinement. The largest source of bending loss is in fact due to mode mismatch – the imperfect mode overlap between straight and bent waveguides. This leads to scattering at the start and end of the fixed-radius bends. It was hypothesized that this can be further reduced, by varying the curvature of the bend continuously rather than abruptly [102]. Regarding the difference between the simulation and measurement results of bending loss, this is because the sidewall roughness measures zero in the simulation tool, which means there is no scattering loss caused by the interaction between the light mode and waveguide sidewall; however, this loss cannot be ignored in real-world measurements.

Subsequently, E-beam lithography became available in the cleanroom. E-beam lithography uses an electron beam to expose the E-beam resist, without the need for a chromium mask. Layouts drawn with L-edit can be directly input into the computer, which controls the electron gun to transfer the layout onto the E-beam resist. Thus, it provides a cleaner environment with higher precision. The deviation of E-beam lithography is smaller than 10nm, which is much smaller than that of the *i*-line MJB4 Mask Aligner in the cleanroom. First, 700 nm of E-beam resist of ZEP was spin coated on the sample. Second, the coated sample was loaded into the chamber for electron exposure. Third, the exposure sample was immersed into the liquid developer of ZED N50 for 60 seconds to do the developing process. Last, the developed sample was rinsed by IPA for about 30 seconds. Strip waveguides were again fabricated on the GON platform with E-beam lithography and the Deep-RIE recipe, and the cross-sectional SEM image and propagation loss are illustrated in *Figure 68*. The selectivity between Ge and E-beam resist is 3:1.

(Caption on the next page)

Figure 68. (a) Cross-sectional SEM image of strip waveguide on GON fabricated with E-beam lithography and Deep-RIE. (b) propagation loss measurement result showing the loss of 2.5 ± 0.2 dB/cm.

4.6 Summary

In this chapter, an optical testing platform was built and the method for waveguide propagation loss measurement is introduced. The standard waveguide fabrication process is described and the influence of lithography and dry etching recipe on the performance of waveguides studied, showing that E-beam lithography and Deep-RIE process can reduce propagation loss prominently, due to a more vertical sidewall and

smaller sidewall roughness. The equation to determine the thickness of Ge for single-mode operation was derived using ray optical theory, followed by modelling the single TE mode strip waveguide on GON using Lumerical FDTD. The simulation results demonstrated that the large refractive index contrast of the GON platform can drastically reduce the radiation loss of bent waveguides, due to better optical confinement, making ultra-compact bent waveguides on GON possible for compact waveguide sensors in Mid-IR sensing applications. The experimental results also indicated that the bending loss of the bent waveguide on GON at radii as compact as $5\text{ }\mu\text{m}$ was only 0.14 dB/bend, which was about 20 times smaller compared to the bent waveguides on GOS at the same radius. In the paper by Chang *et al.* [34], bending loss was 0.12 dB/bend at a $115\text{ }\mu\text{m}$ radius. This bend loss is very similar to the result of 0.14 dB/bend on the GON platform, at a much smaller radius of bend at $5\text{ }\mu\text{m}$, which is 23 times smaller. Thus, the ultra-compact bent waveguide with low loss can be realized on the GON platform. This is a large improvement in the size of bent waveguides, which allows people to design an MZI, micro-ring resonator, or waveguide sensor with a much more compact size.

Chapter 5 Waveguide sensor for Mid-IR sensing application

Mid-infrared (Mid-IR) photonics has recently drawn much attention, owing to its promising applications in next-generation communications, sensing, imaging and so on [15, 22, 46, 103-105]. As far as sensing applications are concerned, Mid-IR provides a simple but effective solution, as the fingerprints of most molecules ($6.7\text{-}20\mu\text{m}$) [106] reside within this range ($2\text{-}15\mu\text{m}$) [107-109]. Based on the interaction of the evanescent field of guiding modes in fibers or waveguides with their surrounding substances, the resulting perturbation of intensity of the mode can be read at the output end of the waveguide. This mechanism can realize both identification of analytes and concentration monitoring at the same time, as different molecules have their respective absorption peaks in the Mid-IR range, and the drop in output intensity is proportional to the concentration of analytes. As people become more health-conscious, greater attention is paid to food consumption. Food additives are inevitable in daily life and they can be found everywhere, such as in condiments, drinks, and snacks. However, illegal food additives and overdosing may cause complications such as cancer. Thus, the food additive must be well-monitored qualitatively and quantitatively in real-time, during the production process in the food industry. Mid-IR sensing will have a major impact on food processing, in addition to environment studies (e.g., detection of hazardous and greenhouse gases), industrial leak detection, process control, medical breath analysis, and many others.

Germanium (Ge) is a suitable material for absorption-based waveguide sensors, given its Mid-IR transparency from 2 to $15\mu\text{m}$, which overlaps perfectly with the fingerprint regions of most molecules; as well as having a large refractive index. Many Ge-based platforms have been made for diversified electronics and photonics applications, e.g. Germanium-on-silicon [110, 111], Germanium-on-insulator (GOI) [112], Germanium-on-chalcogenide (GOC) [41], and Germanium-on-silicon nitride (GON) [113].

5.1 Spiral Waveguide Sensor on GON

The most commonly used platform—silicon-on-insulator (SOI), is unsuitable for Mid-IR sensing application, since the material loss of the buried oxide layer becomes significant at $3.8 \mu\text{m}$ and beyond [114]. Instead, there are two commonly-explored methods: bottom cladding replacement or using a new stack of Mid-IR transparent materials. To replace SiO_2 , many candidates such as suspended silicon [115-117], silicon-on-sapphire [26, 73, 118], and silicon-on-porous silicon [119] have been studied. Examples of new material stack include germanium-on-silicon (GOS) [11, 120], germanium-on-SOI [121], III-V semiconductors [122, 123], and the newly-reported germanium-on-glass [124]. Substantial efforts have been made to extend the operation wavelength to the Mid-IR range. However, the replacement of the bottom cladding usually requires additional steps to remove the bottom cladding layer, like wet etching to remove SiO_2 to form suspended silicon, and new material stacks like germanium-on-silicon, which nevertheless does not provide a high core-clad index contrast ($\Delta n = 0.6$ for Ge/Si at $3.73 \mu\text{m}$), thus making it difficult to realize compact waveguide sensors. In order to take advantage of the wide transparency of germanium, and also to provide a large core-clad index contrast, a new germanium-based platform—germanium-on-silicon nitride (GON)—was fabricated for Mid-IR sensing applications with a refractive index contrast ($\Delta n = 2.1$) close to that of SOI, as described beforehand. This enables bent waveguides to achieve low bending loss even at small radii.

According to equation (36) described below [125], the numerical density of molecules per unit volume is inversely proportional to the effective length of the waveguide and the absorption cross-section of the evanescent field:

$$N_{\min} = (dI / dI_0) / S(\nu)L \quad (36)$$

where dI/dI_0 is the fractional change in light intensity that can be detected, L is the effective absorption path length, and $S(\nu)$ is the absorption cross-section of the evanescent field. As can be seen from equation (36), the longer the effective absorption

path length, the lower the numerical density that can be detected, thus resulting in higher sensitivity. For effective analysis, the length of the waveguide should normally be several millimeters or even longer. In both ref [10] and [12], straight strip waveguides were used in the sensor component, to demonstrate the feasibility of using waveguides for absorption-based sensing applications. However, the straight waveguides would consume a large dimension on the chip in order to achieve a relatively higher sensitivity. If bends are introduced in the waveguide, the size of the sensor can be further reduced, making it more compact and thus more compatible with lab-on-chip applications. The refractive index contrasts of GOS and SOS are smaller than that of SOI, resulting in a higher bending loss, which makes these two platforms unsuitable for designing compact spiral waveguides for sensing applications. A high evanescent field fraction can also improve sensitivity, but may cause high propagation loss. Thus, the GON wafer is a suitable candidate to design and demonstrate compact spiral waveguides for sensing applications. A fluidic chamber is integrated with spiral waveguides to demonstrate the detection of isopropanol and acetone, two commonly-used organic solvents in the semiconductor industry.

Lumerical MODE SOLUTION was used to determine the cross-sectional dimension of the spiral waveguides on the GON platform. In order to extend the evanescent field fraction to give a higher sensitivity, waveguides with a height of $1.5\ \mu\text{m}$ and a width of $1\ \mu\text{m}$ for single TE mode [124] were designed. In addition, waveguides on the GOS platform with a height of $1\ \mu\text{m}$ and a width of $2\ \mu\text{m}$ for single TE mode were also designed for comparison, to obtain a similar propagation loss as the waveguides on GON. According to the simulation results, the evanescent field fraction of waveguides on GON (12.8%) was about three times larger than that of GOS (4.4%) at the wavelength of $3.73\ \mu\text{m}$. The mode profiles of GON and GOS are shown in the inset of *Figure 70(a)*. Since the evanescent field of waveguides on GON was extended, simulations were done to determine the distance between two adjacent waveguides to prevent directional coupling, and the results are shown in *Figure 69*.

Figure 69. The guiding mode profile in two adjacent waveguides with the gap between them of (a) 100nm; (b) 700nm; (c) 1 μ m.

When the gap between two adjacent waveguides was 100nm, there is a strong coupling between them, as shown in *Figure 69(a)*. When the gap was increased to 900nm, some cross-talk can be observed between both waveguides. On the other hand, no coupling was observed when the gap was 1 μ m, as the guiding mode is well confined to the left waveguide, hence no power is transmitted into the right one. Thus, the gap between two adjacent waveguides in the spiral waveguide should be at least 1 μ m.

The fabrication of these waveguides on GON and GOS was carried out using E-beam lithography and Deep-RIE respectively. The etching profile of waveguides on GON is

shown in *Figure 70(b)*. The propagation losses of waveguides on these two platforms were measured using the cut-back method and the results are shown in *Figure 70(a)*.

Figure 70. (a) The cross sectional dimension of waveguides on GON is 1 μm width and 1.5 μm height; while the dimension of waveguides on GOS is 2 μm width and 1 μm height. The propagation losses of waveguides on GOS and GON are $7.28 \pm 1.26 \text{ dB/cm}$ and $7.86 \pm 0.7 \text{ dB/cm}$ respectively. The insets show the mode profiles of waveguides on the two platforms. (b) The cross-sectional SEM image of the waveguides fabricated on GON platform.

The propagation loss of waveguides on GON was 7.86 dB/cm, which is almost the same as the loss of waveguides on GOS (7.28 dB/cm), even with a higher evanescent field fraction. This is mainly because of the higher core-clad index contrast of the GON platform, resulting in better mode confinement at the core/under-clad interface than that of the GOS wafer. Besides, the misfit dislocations were removed through the layer

transfer process, resulting in a lower scattering loss. The radii of the bent waveguides were determined by MODE SOLUTION simulation. The radius of $30\text{ }\mu\text{m}$ was chosen for bent waveguides on GON, and $200\text{ }\mu\text{m}$ for GOS, as the bending losses of the spiral waveguides on these two platforms are comparable as well as negligible at these two respective radii. This demonstrates one of the advantages of the GON platform, namely that, a large core-clad index contrast allows for a compact waveguide design suitable for lab-on-chip applications.

Spiral waveguides were designed on GON with a size of $260\text{ }\mu\text{m}$ by $260\text{ }\mu\text{m}$, and a total length of 4.02 mm such that the total loss measured 3.14 dB , which is acceptable to our testing system. The gap between two adjacent waveguides was $3\text{ }\mu\text{m}$, which was large enough to prevent directional coupling. The top view of the spiral waveguide on GON, as seen from an optical microscope, is shown in *Figure 71*.

Figure 71. The top view of the spiral waveguide sensor on GON platform.

Spiral waveguides of the same length were also fabricated on the GOS platform, where the radius of the bent waveguide was larger than that of waveguides on GON, resulting in a much larger total area ($600\text{ }\mu\text{m} \times 600\text{ }\mu\text{m}$). A fluidic chamber made out of PDMS [126] was fabricated and integrated with the sensor chip. The absorption spectrum of cured PDMS was reported by Cai *et al.* [127] and shown in *Figure 72*.

Figure 72. The absorption spectrum of cured PDMS in IR range [123].

It can be seen from Figure 72 that cured PDMS has an absorption peak within 3 to 4 μm , centered at 3.4 μm . The operation wavelength used for isopropanol sensing was 3.73 μm , where the absorption from PDMS can be ignored, as the absorbance of PDMS is close to 0 at 3.73 μm , as shown in Figure 72. The schematic of the fabrication process flow of the PDMS chamber is shown in Figure 73 and described below.

Figure 73. (a) 3D printing mold according to waveguide design; (b) Pour liquid PDMS in mold and then degas and cure; (c) peel it off after solidification; (d) bond it to waveguide chip; (e) introduce inlet and outlet channels; (f) inject liquid in chamber for sensing.

Firstly, PDMS in the liquid phase was poured into a 3D-printed hard mold and put into a vacuum desiccator to degas the PDMS by eliminating air bubbles, as shown in *Figure 74(a)*. Secondly, the PDMS was cured on a hot plate for 15 mins under 90°C for solidification. Thirdly, the PDMS was gently peeled away from the hard mold, followed by punching tube holes for liquid transportation. Finally, the PDMS chamber was bonded onto the required substrate, using liquid-phase PDMS itself to seal the edges of the chamber. *Figure 74(f)* is an example of liquid being handled in the PDMS chamber. Once the PDMS chamber was ready, the sensor chip was bonded with the fluidic chamber and a syringe pump system for liquid sample handling was also integrated with the chamber, as shown in *Figure 75*.

Figure 74. The fabrication process of PDMS fluidic chamber.

Figure 75. The sensor chip bonded with fluidic chamber.

The liquid used to demonstrate the feasibility of the spiral waveguide sensor was isopropanol, also known as IPA. This organic liquid is commonly used in the semiconductor industry for cleaning wafers, and has an absorption coefficient of about 144 cm^{-1} at $3.73 \text{ }\mu\text{m}$ [128]. Acetone was used as the solvent and its absorption is negligible, as the transmittance of acetone is about 100% at this wavelength. Different volumetric concentrations of IPA mixed with acetone solution were prepared before measurement. The absorption (in dB), A , induced by IPA in the fluidic chamber, was calculated by considering the ratio of light intensity transmitted through the waveguide surrounded by pure acetone (I_{solvent}) and by a solution of IPA and acetone (I_{analyte}), defined as:

$$A = 10 \log_{10} \left(\frac{I_{\text{solvent}}}{I_{\text{analyte}}} \right) \quad (37)$$

The sensor chip was mounted on a XYZ translational stage with a vacuum chuck. A light source, with a $3.73 \text{ }\mu\text{m}$ wavelength from a rapid scan CW-Pulsed quantum cascade laser from DAYLIGHT SOLUTIONS, went through a polarization controller, which was then butt-coupled in and out of the spiral waveguide using a pair of single mode ZrF_4 fibers from Thorlabs. TE mode measurements were used, similar to the

previous simulations. The same waveguide was used throughout the experiment to eliminate any non-uniformity issue associated with waveguide intrinsic loss. For each measurement, a 0.4 ml solution was injected with different volumetric concentrations of IPA into the chamber and removed with a syringe after a successful measurement. The chip was realigned for maximum output before subsequent measurements. The output signals were measured with a high sensitivity InSb photodetector (-40dB) from HORIBA. The same process was repeated on the waveguide sensor on the GOS platform as a benchmark. In the current model adopted, the noise level of the testing platform is given as follows [129] :

$$C_{\min} = \frac{\sqrt{2}}{L\alpha\eta} \sqrt{\left(\frac{\Delta I_0}{I_0}\right)^2 + \left[\frac{\sigma_j}{RI_0 \exp(-PL)}\right]^2} \quad (38)$$

where R is the photodetector responsivity, I_0 is the input laser intensity coupled into the waveguide, P is the intrinsic waveguide loss not considering the absorption of analyte, L represents the effective absorption length of the waveguide, α is the absorption coefficient of pure analyte, η is the evanescent field fraction in the solution, and σ_j denotes dark current noise from the detector. The terms in equation (38) within a square root correspond to two major noise sources: input laser power fluctuation and photodetector noise at the output. The latter term is of influence only when the analyte solution has strong absorption or is highly concentrated, as in the case of heavy water, with an absorption coefficient of 18000 cm^{-1} at around $4 \mu\text{m}$ [73]. In the current system, the former term, fluctuation of laser input power, is about 6% ($\sim 0.26\text{dB}$) of optical power, effectively passing through the solution. Therefore, the theoretical lowest concentration of IPA in a mixture solution of IPA and acetone that can be sensed by the spiral waveguides is 5% for the GON platform and 16% for the GOS platform respectively, as the absorption for even lower concentrations of IPA in acetone cannot be distinguished from the noise in the system. The experimental measurement results at different concentrations of IPA in IPA-acetone mixtures are shown in *Figure 76*.

Figure 76. Absorption of spiral waveguide sensor at different IPA-acetone mixtures. The lowest volumetric concentration of IPA that can be measured by the sensor on GON is 5% while for GOS platform it is 16%. Inset: Waveguide output for laser scan with acetone and IPA as the analytes.

The lowest volumetric concentration of IPA measured by the sensors on the GON and GOS platforms were 5% and 16% respectively. The experimental measurement results are comparable with the theoretical values, which verify that the performance of sensors built on the GON platform is superior to that of GOS, due to its higher evanescent field fraction. This high evanescent field fraction does not greatly sacrifice the propagation loss of the waveguide on GON, owing to the large core-clad index contrast, as well as a transferred Ge layer free of misfit dislocations. This validates the feasibility of using GON as a high-performance sensing platform. Lastly, the laser scan (3.65 to 3.9 μm) results, carried out for the spiral waveguide sensor on GON immersed in 100% acetone / 100% IPA, are shown in the inset of Figure 76. It is observed that the loss remains constant over most of this range. This is expected, as the absorption spectrum of IPA is broad around this wavelength region [128]. For analytes like IPA or acetic acid [23], whose absorption spectrums are wide, this spiral waveguide can work as a broad band sensor. This chip can also be used for trace gas sensing applications,

with its integration to a proper gas sample handling and transporting system, as many gas molecules have their absorption peaks in the Mid-IR range [24].

A spiral waveguide sensor was fabricated on the GON platform and was demonstrated to have better performance than the GOS platform with nearly the same propagation loss. A high-quality Ge layer free of misfit dislocations, and a structure with high core-clad index contrast was realized, by the wafer bonding and layer transfer technique. The GON platform assisted in designing a waveguide with a high evanescent field fraction to improve absorption-based sensitivity, while still keeping propagation loss acceptable. The lowest concentration of IPA in acetone, measured in these experiments, was 5% for GON versus 16% for GOS, demonstrating the feasibility of using GON as a compact, effective, and accurate platform for Mid-IR sensing applications. The sensors fabricated on the GON platform can be integrated with a photodetector and a read-out circuit for lab-on-chip applications, which can potentially be used in industrial leak detection, process control, and medical analysis, among others.

5.2 Pedestal Waveguide Sensor on GOI

The germanium-on-insulator (GOI) platform has drawn much interest in recent years, due to its potential for future Ge-based CMOS electronic and photonic integration. Thus, it would be promising to fabricate a waveguide sensor on the GOI platform to be integrated monolithically with a photodetector, and even a light source in the future, to realize a sensor on the chip. The TM mode is preferred for sensing applications because it has a larger evanescent field fraction (EFF) than the TE mode and thus a higher sensitivity. However, the buried oxide is lossy in the Mid-IR range, especially for the TM mode, due to poor vertical confinement. In order to resolve this limitation, a method to undercut the buried oxide to reduce loss has been proposed, and this structure is called the pedestal waveguide. The loss reduction of the SOI pedestal waveguide with a specified guiding mode for deep Mid-IR has been theoretically studied by He *et al.*[130], and the sensing applications of pedestal waveguides has also been explored by Lin *et al.* [70]. However, there remains very limited discussion on

waveguide performance, together with an experimental investigation of the waveguide and its application in sensing.

In this section, the modelling of pedestal waveguides built on a scalable GOI platform developed in-house is first discussed. The influence of notch width of the buried oxide layer on the EFF of fundamental TE and TM is studied. Pedestal waveguides on GOI were fabricated by undercutting the buried oxide with the wet etching process, and propagation loss of the waveguide for both TE_{00} and TM_{00} was measured. Acetic acid was chosen to be the analyte for the sensing application, given its strong and broad absorption peak between $3\text{-}4\mu\text{m}$ and it being a common chemical in daily life and regularly used in the food industry. The sensing results confirm that the extended EFF of the TM_{00} mode can improve its sensitivity.

First, single mode waveguides were designed on the GOI platform at $3.81\ \mu\text{m}$ wavelength using Lumerical MODE SOLUTION, and the mode profiles are shown in *Figures 77(a) and (b)*. According to the simulation results, the fundamental TM mode cannot be supported when the thickness of the Ge layer is smaller than $1\ \mu\text{m}$, and the higher TE mode will be activated when the width of the waveguide is larger than $2.2\ \mu\text{m}$. Thus, the cross-sectional size of the waveguide was set to be $2\ \mu\text{m}$ wide and $1\ \mu\text{m}$ high. The thickness of the buried oxide was set to be $500\ \text{nm}$, based on the actual GOI wafer that was fabricated previously. Furthermore, the influence of the notch width, which connects the buried oxide to the Ge layer on the EFF in both modes, was studied. As described in equation (36), the minimum concentration of the analytes that can be detected is inversely proportional to the effective length of the waveguide and the evanescent field fraction of the guiding modes. Thus, increasing the length of the waveguide is a straightforward way to improve sensitivity, defined as the lowest volumetric concentration of the analyte as per the spiral waveguide designed in section 5.1. However, this can adversely exacerbate propagation loss. Another possible method is to enhance the EFF to improve sensitivity, at the same time keeping the compact size of the waveguide sensor. The cross-sectional size would affect the EFF, as the evanescent field will be extended when the cross-sectional size shrinks. However, the

shrinking of the cross-sectional size will make the guiding mode more sensitive to sidewall roughness, resulting in more undesired loss. Thus, undercutting the buried oxide is an optimal choice to extend the EFF, as well as to reduce loss, as the oxide is opaque in the Mid-IR range beyond $3.8\ \mu\text{m}$. The notch width of the buried oxide was increased from 200 to 2000nm at an incremental step of 200nm. The EFF of the pedestal waveguide, that can directly interact with analytes surrounding the waveguide, is plotted as a function of the notch width in *Figure 77(c)*. The EFF at different widths and heights of waveguides was also simulated, and the result indicates that a smaller cross-sectional size will lead to a higher EFF.

Figure 77. (a) mode profile of the fundamental TE mode (TE_{00}); (b) mode profile of the fundamental TM mode (TM_{00}) and (c) the EFF of both modes as a function of the notch width; (d) the EFF at different width and height of waveguides.

As can be seen from the simulation results, the EFF of the fundamental TM mode (TM_{00}) is more than two times (9.54% vs 4.14%) larger than that of the fundamental TE mode (TE_{00}) at the notch width of 200 nm. As the notch width decreases, the EFF

of TE_{00} also decreases, because the waveguide becomes an air-clad waveguide, noting that the refractive index of air is smaller than that of oxide. Hence, the larger refractive index contrast gives a better confinement for the TE_{00} mode. On the contrary, the EFF of TM_{00} increases as the notch width decreases, because this rectangular-shaped waveguide has a poor vertical confinement for TM modes; also that the evanescent field of TM modes is larger than that of TE modes originally. The removal of the buried oxide exposes more of the evanescent field of TM_{00} , contributing to the increase of the EFF for TM_{00} .

In order to demonstrate that the TM_{00} can enable a higher sensitivity, waveguides were fabricated on a GOI platform (fabricated in-house) with electron-beam lithography and deep-reactive ion etching (Deep-RIE). The GOI wafer was fabricated by the collaborator from Singapore-MIT Alliance for Research and Technology (SMART). The fabrication process and characterization of the bonded GOI wafer can be found in reference [10]. The waveguides were bent to form a spiral shape with a size of $850\ \mu m \times 850\ \mu m$ and a total length of 5 mm for saving the area on the chip, similar to the spiral waveguide on GON introduced in the previous section. The radius of the bend was $100\ \mu m$, and the bending loss can be disregarded, according to the simulation results. Besides the spiral waveguide sensor, a set of waveguides with different lengths were also fabricated for propagation loss characterization, using the cut-back method. After the waveguides were patterned and etched, the chip was immersed in diluted hydrofluoric acid, also known as Buffered Oxide Etchant (BOE), for undercutting the buried oxide to form the pedestal structure. The etching rate for oxide exposed was 2 nm/s. Since the wet etching process is isotropic, the etching time was set at 450 s to remove 900nm oxide on either side of the notch. After etching, the sample was inspected under SEM, and the cross-sectional SEM image is shown in *Figure 78*.

Figure 78. The cross-sectional SEM image of pedestal waveguide after BOE etching.

After immersing the sample in BOE solution for 450s, only a little oxide underneath the Ge was removed, indicating that the etching rate changed. Several trials were carried out to define the wet etching rate, as the etch rate of BOE will slow down even when trace amounts of BOE are able to enter the oxide underneath the Ge layer. Finally, a notch width of around 200 nm was realized and a cross-sectional scanning electron microscope (SEM) image of the pedestal waveguide structure following BOE etching is shown in *Figure 79*.

Figure 79. Cross-sectional SEM image of the fabricated pedestal waveguide on GOI platform.

The sensor chip was mounted on the XYZ translational stage with a vacuum chuck on the optical table. The vacuum chuck and optical table ensure that there are no vibrations disrupting the results when a liquid droplet is cast on the chip. The measurement was conducted under room temperature (24°C) which was stable and controlled by the central air conditioner. The influence of temperature on sensitivity was not studied this time, as there was no temperature-regulating component integrated on the stage. The characterization of the loss performance of the waveguide was done by employing the cut-back method, and the result was 3.6 dB/cm for the TM_{00} mode and 2.5 dB/cm for the TE_{00} mode. Then, the pedestal waveguide sensor was mounted on the stage for measurement, with the same waveguide used throughout the experiment to eliminate any non-uniformity issues associated with waveguide propagation loss. In the case of TE_{00} , confinement was improved; thus the loss is smaller than that of TM_{00} and was mostly caused by sidewall roughness scattering. The sidewall roughness of Ge waveguides can be smoothed by a quick dipping in H_2O_2 [131]. The analyte used to demonstrate the sensing application of pedestal waveguides was acetic acid (CH_3COOH). This organic acid is commonly used in the food industry for condiments and food additives. It has a broad absorption peak, ranging from 3 to 4 μm , with the tip residing at $\sim 3.45 \mu m$.

Figure 80.(a) The transmittance spectrum of acetic acid solution in IR range. (b) the enlargement of transmittance spectrum from 3.4 to 4 μm [132].

However, due to the light source in the optical testing system only being tunable from 3.65 to 3.90 μm , the wavelength of 3.81 μm was chosen as the probing wavelength, at which the second strong absorption peak of acetic acid resides. Water was used as the solvent as its absorption is negligible, and whose transmittance is about 100% at the wavelength of 3.81 μm . The absorption (in dB), A , induced by the acetic acid droplet on the sensor chip, was calculated by considering the ratio of light intensity transmitted through the waveguide surrounded by pure water, and by a solution of acetic acid and water similar to equation (37). For each measurement, a 0.2 ml amount of solution was injected with different volumetric concentrations of acetic acid onto the chip. The chip was demounted after a successful measurement, followed by drying and cleaning, and was then mounted on the vacuum chuck again for realignment to ensure a maximum output signal for the subsequent measurements. The sensing results are demonstrated in *Figure 81*.

Figure 81. Sensing result using droplet test. The lowest concentration detected for TM_{00} is 0.1% while for TE_{00} it is 0.25%.

Based on the testing results, the lowest concentration that can be measured is a 0.1% volumetric concentration of acetic acid in water, using the TM_{00} mode, which equals to 3000ppm (parts per million). For concentrations smaller than 0.25% using the TE_{00} mode and concentrations smaller than 0.1% using the TM_{00} mode, the decrease in output is too small to be differentiated between the absorption of acetic acid and the fluctuation of the testing system, as they are lower than the noise floor of the system. According to equation (36), the different sensitivities between the TE and TM mode are caused only by the different evanescent field fractions, since the length of the waveguide stayed consistent throughout the whole testing process. Additionally, the loss difference between TE_{00} and TM_{00} makes the pedestal waveguide useful for lab-on-chip applications when combined with the mode splitter and converter, as the low-loss TE mode can be used for signal transmission and the TM mode for sensing application. In order to further improve the sensitivity of waveguide sensors for practical use, special structures like slot waveguides could be incorporated, as most of the light will be confined within the air-gap slot, which makes the field fraction able to

directly interact with substances as large as 30% [133]. Compared to our pedestal waveguide with 9.5% EFF, this is three times higher, resulting in a much lower concentration able to be sensed. The primary limitation with the slot waveguide is its propagation loss, as most of the light is exposed in the narrow air-gap, which makes it very sensitive to the imperfections of the waveguide. The loss reduction of the slot waveguide with the application of advanced lithography techniques, dry etching, and surface passivation needs to be studied in order to provide an adequate sensitivity for Mid-IR sensing applications.

Pedestal waveguides were designed and fabricated on in-house fabricated GOI platforms. The sensing application of the pedestal waveguide was studied, and the lowest concentration of 0.1% acetic acid in water demonstrated. The sensitivity can be further increased by extending the evanescent field fraction using specially-designed structures, like slot waveguides. Besides acetic acid, there are many other molecules which also have absorption resonance at this sensing wavelength such as methane, nitrogen dioxide, water, and sulfur dioxide. By choosing proper probing wavelengths, different gases and liquids can be identified and quantified. This simple but effective method can make a substantial impact on environmental, medical, and food industry applications.

5.3 Slot Waveguide Sensor on GON

As mentioned in section 5.2, the sensitivity of a sensor can be improved by increasing the evanescent field. The slot waveguide is a good candidate for enlarging the evanescent field, as most of the light will be confined within materials with a low refractive index. Unlike light guided by total internal reflection in a regular planar waveguide, light in this case can be guided and enhanced in the low-index material, as reported by Almeida [133]. According to Maxwell's equation, in order to satisfy the continuity of the normal component of electric flux density for a high-index contrast interface, the corresponding electric field must undergo a large discontinuity with a much higher amplitude in the low-index portion, meaning that the amplitude of the electric field in the low-index material would be n_H^2/n_S^2 (n_H being the refractive index

of high-index material and n_s being the refractive index of low-index material) times higher than that of in a high-index material. The geometry of the slot waveguide is usually referred as a low-index slot embedded between two high-index slabs. This kind of structure has been widely used in photonics for high-Q optical resonators [134], label-free biosensing with a slot waveguide-based ring resonator [135, 136], absorption based sensing [71], among others. However, the operation wavelengths in these references are all within $3.8 \mu m$, which is mainly restricted by the platform (SOI) used. Absorption-based sensors require the operation wavelength to be extended to even longer regions, where most molecules have their absorption peaks. Thus, the slot waveguide was modelled and fabricated on the GON platform for longer wavelength sensing applications in the Mid-IR range. As mentioned before, the geometry of slot waveguide is simple, being only a low-index slot embedded between two high-index slabs. However, the mode transmitted in the slot region is quite different from the mode propagating in the strip waveguide; a converter must be employed to convert the mode in and out of the slot region. During modelling, the Y-junction was used as the converter and the influence of all parameters on the transmission of light in the slot region was studied. The schematic of the Y-junction and slot waveguide is shown in *Figure 82*.

Figure 82. Schematic of Y-junction converter and all parameters related.

As shown in *Figure 82*, W is the width of strip waveguide, h the height of the structure, d the distance between taper tip to strip waveguide, L_{taper} the length of taper, w the width of high-index slab, and g the width of slot. In the simulation, W was set to be $2\text{ }\mu\text{m}$ and h was $1\text{ }\mu\text{m}$ for single-mode transmission at the wavelength of $3.9\text{ }\mu\text{m}$, given that the largest wavelength of the tunable laser in the optical testing platform is $3.9\text{ }\mu\text{m}$. L_{taper} was set to be $10\text{ }\mu\text{m}$, w was set to be 300 nm and g was 100 nm , denoting the start point, which were then swept to find the largest transmission of light in the slot region. The simulation was done by Lumerical MODE SOLUTION and the results are shown in *Figure 83*.

Figure 83. (a) a top-view image of light transmission in slot waveguide. (b) the distribution of effective refractive index of electric field in slot waveguide.

According to the simulation, the transmittance of light in the slot region is about 40%, and the coupling efficiency of the Y-junction converter is about 94%. Afterwards, the widths of the slot and the high-index Ge slab were swept to find the optimal condition. The simulation results are illustrated in *Figure 84*.

Figure 84. Transmittance in slot region as a function of the width of high-index Ge slab at (a) different width of slot and; (b) different height of the structure.

When the width of the high-index Ge slab was swept at three different slot widths, the height h was fixed at $1\ \mu\text{m}$. The simulation result demonstrates that the largest transmittance appears at $g=100\ \text{nm}$. When the width of the slot increases or decreases, the transmittance drops, indicating that the optimal number of the slot to be $100\ \text{nm}$. Similar to the height optimization, the gap g was fixed at $100\ \text{nm}$ for the second sweep and the optimal height h was found to be $1\ \mu\text{m}$. According to the simulation results, the optimal parameters for largest transmittance (48%) in the slot region is $w=350\ \text{nm}$, $h=1\ \mu\text{m}$, and $g=100\ \text{nm}$. Once the parameters were determined, the slot waveguides were fabricated using E-beam lithography and Deep-RIE. The SEM images of the fabricated slot waveguides are illustrated in *Figure 85*.

Figure 85. (a) top-view of Y-junction converter. (b) bend part of the slot waveguide. (c) cross-sectional image of the slot waveguide. (d) tilted Y-junction converter.

The fabricated slot waveguides were characterized on the testing platform, showing high propagation loss, because the light confined in the slot region is very sensitive to

sidewall roughness and fabrication imperfections, such as the notch in *Figure 85(d)*. The measurement result is shown in *Figure 86*.

Figure 86. Loss measurement result of slot waveguide.

The propagation loss of the slot waveguide was found to be 2.39 dB/mm, which requires further reduction for practical sensing applications. The method of surface passivation by atomic layer deposition was reported by Alasaarela *et al.* [137]. They claimed that surface roughness can be drastically reduced by atomic-layer depositing 30nm TiO₂ on the slot waveguides on the SOI platform, reducing loss to 7 dB/cm. As can be seen from the loss measurement result, the experimental data points are widely scattered from the fitting line, indicating a lack of consistency in the measurement process. This was due to imperfections occurring during fabrication, as the critical size of the slot waveguides on GON is extremely small, similar to the taper tip of the Y-junction. The photoresist on the taper tip part may have been over-developed or could not bear the dry etching process, causing over-etching as shown in *Figure 85(d)*. These imperfections may cause inconsistencies in coupling efficiency, thus leading to the departure of the experimental data from the straight line. The fabrication process will also have to be optimized for a better fabrication result. Furthermore, the sensing

application of the slot waveguide sensor on the GON platform has yet to be demonstrated.

5.4 Summary

Waveguide sensors for Mid-IR sensing application were described in this chapter. For absorption-based sensing, there are two methods to improve sensitivity: either by enlarging the total length or extending the evanescent field fraction. Spiral waveguide sensors were fabricated on the GON platform for increasing the contact area between analytes and light, in order to improve sensitivity. A fluidic chamber made of PDMS was fabricated and bonded onto the sensor chip for liquid sample transportation and handling, and the spiral waveguide sensor demonstrated a 5% sensitivity to IPA in acetone solution. The pedestal waveguide sensor was realized on the GOI platform, as the oxide layer under the Ge can be undercut to extend the evanescent field fraction for the fundamental TM mode. The results indicated that the TM mode had 0.1% sensitivity to acetic acid in water, which is higher than that of the TE mode. The slot waveguide was proposed for high sensitivity application, due to its high transmittance of light in the air slot region, where analytes can directly interact with the guiding light.

Chapter 6 Conclusions and Future Work

6.1 Conclusions

Mid-IR photonics has attracted much research interests in recent years due to its enormous potential for next-generation communication and sensing applications. Photonic integrated circuits would form the mainstream for future photonics research and the monolithic integration between photonics and electronics for compact, cost-effective, and reliable chips are meaningful to break the restriction of Moore's law to the electronics industry and to commercialize photonics chips based on well-established industrial foundries.

A novel platform—germanium-on-silicon nitride (GON) was proposed for Mid-IR photonics, as it has a wide transparency window of up to $7.5\ \mu\text{m}$ in the Mid-IR range, as well as a large core/clad refractive index contrast, which can provide strong confinement for modes passing through a bent waveguide. Furthermore, the SiN_x layer is also transparent in the near-IR range, which can be used as a waveguide to connect the abovementioned Ge modulator and Ge photodetector to realize a lab-on-chip system, by engineering the thickness of the SiO_2 bonding layer under the SiN_x . GON was realized by the wafer bonding and layer transfer technique. In order to do this, the bonding criterion has been carefully studied as wafer-level bonding has many critical requirements. The stress compensation method has been developed to solve the film cracking problem and to also ensure that the wafer bow is smaller than $25\ \mu\text{m}$. The origin of thermal bubbles has been studied and solved, by employing a thick SiO_2 layer as the bonding layer. The final bonded GON wafer had no thermal bubble, void, or particle found under IR imaging inspection. The quality of the Ge layer showed no degradation during the whole bonding process, which was confirmed by HRXRD and some tensile strain residue in the Ge layer, due to the thermal expansion coefficient mismatch between the Ge and Si substrates.

The optical testing platform was set-up for waveguide loss characterization and sensing testing within the Mid-IR wavelength range from 3.65 to $3.90\ \mu\text{m}$. The testing platform is compatible for both grating and end-fire coupling methods. A lock-in amplifier was

employed to deal with the relatively low sensitivity of the optical detector at the output end of the system. The light source can be modulated by the chopper in order to add a carrier wave, then identified by the lock-in amplifier to amplify the signal, as well as to rule out background noise. The noise level of the system was as low as 0.23 dB when sensing testing was conducted. All the components of the testing system were connected by *Labview* and controlled by a computer, therefore, highly efficient measurements can be realized by this platform.

A standard fabrication process flow for waveguides was developed. By using optical lithography and the RIE dry etching process, the critical size of 2 μm can be realized. The rib waveguides fabricated on the GOS platform demonstrated a propagation loss of 6.85 dB/cm, while the loss was reduced to only 2.65 dB/cm by employing the Deep-RIE dry etching recipe, indicating that the fabrication process has a large influence on the performance of devices. Later, an E-beam lithography was introduced in the cleanroom, with it being able to realize the critical size of 50 nm. The surface of the photoresist after E-beam lithography and developing was quite clean and sharp, reducing the propagation loss of the strip waveguide on GON from 3.3 dB/cm to 2.5 dB/cm. The ultra-compact bent waveguide was modelled in Lumerical FDTD, showing a much lower bending loss at a radius of only 5 μm when compared to the bent waveguide on the normal GOS platform. After fabrication, the bent waveguides on GON and GOS were characterized respectively, and the bending loss for GON was only 0.14 dB/bend at the radius of 5 μm , while the loss for GOS was 2.54 dB/bend at the same radius, a value 20 times higher. Thus, the GON platform demonstrated its capability of realizing ultra-compact low-loss bent waveguides at the Mid-IR range.

Absorption-based waveguide sensors for Mid-IR sensing application were designed, fabricated and tested. In order to improve the sensitivity of the waveguide sensor, two common methods were employed: increasing the total length of waveguide to increase the contact area between analytes and the waveguide, as well as extending the evanescent field fraction. A spiral waveguide sensor was fabricated on the GON platform in order to increase the total length, while maintaining a relatively compact

size by bending the straight waveguide into a spiral shape. The evanescent field fraction was also extended, by shrinking the lateral size of the waveguide for the TE mode. It demonstrated a 5% sensitivity to IPA in acetone solution at the wavelength of $3.73\ \mu\text{m}$. IPA has a absorption peak located at this wavelength, while acetone is about 100% transparent. Thus, this spiral waveguide can be used to distinguish IPA from acetone solution in semiconductor industries, as these two solutions are frequently used. 5% is not low enough for practical applications, like quantifying the concentration of IPA in real-time. Methods must be devised to improve its sensitivity, namely the use of the slot waveguide. A pedestal waveguide on the GOI platform was realized by undercutting the buried oxide layer of the GOI wafer, in order to further extend the evanescent field fraction for the TM mode, as it has poor confinement in the vertical direction. Given that acetic acid has a strong absorption peak at $3.81\ \mu\text{m}$, this pedestal waveguide was used to demonstrate acetic acid sensing. The evanescent field fraction of the TM mode is about 2 times larger than that of the TE mode, and the sensing results show that sensitivity to acetic acid is 0.10 % for the TM mode while being 0.25% for the TE mode. This volumetric concentration equals to 3000 ppm of sensitivity, which is much better than the result of the spiral waveguide sensor, but still not good enough for practical applications, where several ppm is required. Therefore, the best solution to improve sensitivity is to use the slot waveguide as the sensor, because light is confined in the low-index slot region, which is normally air. This property makes it ultimately sensitive to the variations in the surroundings, as the light confined in the slot region can directly interact with analytes. However, the propagation loss is quite high for fabricated slot waveguides. Fabrication process optimization and proper surface passivation are necessary to reduce coupling inconsistency; while low surface roughness is necessary to reduce propagation loss due to surface scattering.

6.2 Future Recommendations

The work reported in this thesis provides a novel platform for Mid-IR photonics, whose transparency window is extended to $7.5\ \mu\text{m}$, and that has a large core/clad index

contrast. Based on this understanding and the experiment results obtained from this study, the following recommendations are proposed for future work:

a. Slot waveguide on GON for Mid-IR sensing applications. This waveguide structure has a congenital advantage in the interaction between confined light and analytes, thus realizing high sensitivity. Firstly, to reduce propagation loss of slot waveguides on GON, surface passivation has to be conducted with the atomic layer deposition of Al_2O_3 . The effect of the Al_2O_3 deposition layer on the loss and transmittance of light in the slot region should be studied, as well as the optimal thickness of the Al_2O_3 deposition layer. As described in reference [71], a microfluidic chamber can be integrated with the sensor chip for better sample transportation and handling. The microfluidic chamber has a higher requirement for this fabrication technique, due to its small size. If a slot waveguide sensor with a relative small propagation loss can be realized and integrated into this microfluidic chamber, it will have a major impact on Mid-IR sensing, because of the combination of high sensitivity and compact size.

b. Besides absorption-based waveguide sensors, dispersion sensing-based microring resonators can be developed on the GON platform. Due to the large core/clad refractive index contrast, microring resonators can have an even smaller size on this platform while maintaining a high Q-factor. Different effects can be introduced to the microring resonator to enhance its sensitivity, such as the Vernier effect [138] and self-interference [67].

c. Since SiN_x is transparent from the near-IR to Mid-IR range, it is a good medium to transmit light of different wavelengths. Ge is a good material for photodetection at the wavelength of $1.55 \mu\text{m}$ and many high-performance Ge photodetectors have been reported [139-141]. It is possible to use this platform to integrate the modulator, waveguide sensor, and photodetector to form a lab-on-chip system. In order to guide the light in the SiN_x layer, the thickness of the SiO_2 bonding layer must be engineered to be thick enough to prevent the leakage of the $1.55 \mu\text{m}$ wavelength to the substrate. Meanwhile, the wafer bonding process cannot be affected by this thickness. This schematic is shown in *Figure 87* below. If the integration of the waveguide sensor and

photodetector can be realized, the sensing information can be transferred into an electrical signal, and thus can be read out either by circumscribing or integrating a read-out circuit, so that the sensing information can be reflected on a display in real-time. Modulators can be realized by applying compressive strain on Ge to modify its bandgap, thus changing its cut-off wavelength, and then integrating with the SiN_x waveguide and Ge photodetector. By adding the SiN_x stressor on Ge, the bandgap of Ge can be engineered [142].

Figure 87. The schematic of waveguide integrated Ge p-i-n photodetector.

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