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Swansea University
Prifysgol Abertawe

Submitted to Swansea University for the fulfilment of MPhil in Electronic and Electrical Engineering

Development of an Optically Transparent Silicon CMOS Technology Platform for Biological Analysis

Nigel Davies

2012

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Summary

The traditional method of analysing biological cells mounted on glass slides is well documented. However, more modern techniques have been developed to allow the automated sorting and isolation of single cells for biological investigation using Lab-On-a-Chip (LOAC) technology. LOAC technology utilises the power of the integrated circuit to allow high cell count sorting and positioning. A disadvantage of the current generation of LOAC devices is that when the cell sorting procedure has been completed the isolated cells then have to be removed to allow more detailed biological analysis.

This thesis concentrates on the research and development of an optical transparent window and corresponding via which extends from the top surface to bottom surface of a silicon wafer. The optical structure would then be integrated onto a LOAC cell positioning platform that would then allow the *in situ* analysis of the cells using optical techniques through the substrate. The top surface would then be available to be used in conjunction with other analytical techniques such as atomic force microscopy (AFM).

The method of selection of suitable silicon compounds and a range of thicknesses, to form an optical window, and the required fabrication techniques to deposit the compounds onto silicon wafer is illustrated. Additionally a dry etch method is identified to enable the construction of an optical via to be etched completely through the silicon wafer. The transmission priorities of the optical widow is characterised and the analysis of a biological cell line using epi-fluorescence microscopy utilising both brightfield and fluorescence transmission techniques is demonstrated.

Declarations and Statements

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

This thesis is the result of my own investigations, except where otherwise stated. Other sources are acknowledged by footnotes giving explicit references. A bibliography is appended.

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Acknowledgements

I wish to thank Dr. Paul Holland, my supervisor for his help, guidance and support during the preparation of this thesis.

I would also like to thank Dr Huw Summers, Dr Mark Holton and Karthik Rajasundaram from College of Engineering at Swansea University for the help and guidance on optical analysis.

Thanks are also due to my employer SPTS Ltd for their support throughout this project.

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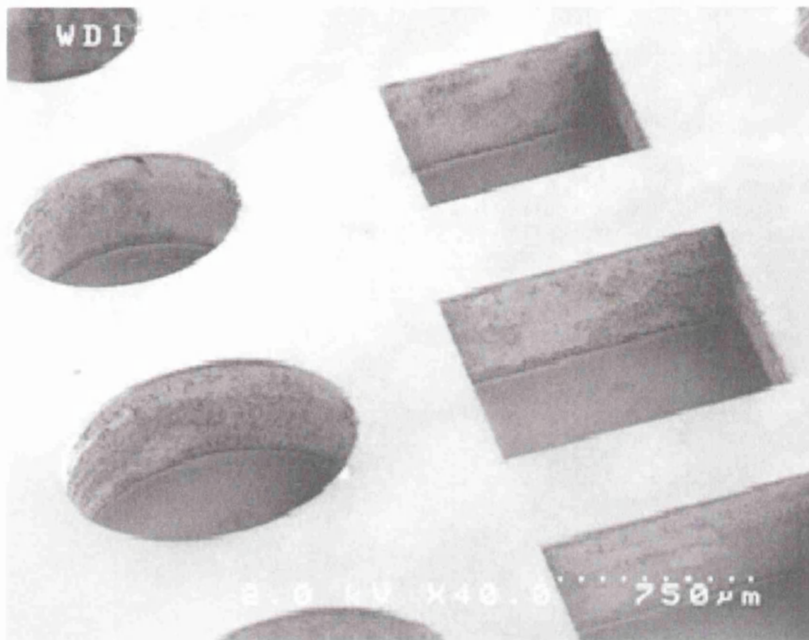
Definitions or Abbreviations

AFM	Atomic Force Microscopy
CMOS	Complimentary Metal Oxide Semiconductor
CMP	Chemical Mechanical Polish
DSE	Deep Silicon Etch
DNA	Deoxyribonucleic acid
DI	Deionised, Commonly used in DI water
EPD	Endpoint Detector
ESC	Electrostatic Clamp
IC	Integrated Circuit
LOAC	Lab-On-A-Chip
MEMS	Micro-Electro-Mechanical Systems
PECVD	Plasma Enhanced Chemical Vapour Deposition.
PVD	Physical Vapour Deposition.
RF	Radio Frequency
RI	Refractive Index
sccm	Standard Cubic centimetres per Minute

SEM	Scanning Electron Microscope
TEOS	Tetraethylorthosilicate
TSV	Through Silicon Via
via	Cylindrical or square hole etched into a silicon Wafer



1. Introduction

One of the most rapidly expanding technologies today is the development of micrometer and sub micrometer mechanical and electronic structures. These structures are already widely used in everyday life, for example air bag sensors used in the automotive industry and the gyroscopic sensors used in mobile phones. Advances in the fabrication techniques of Complementary Metal Oxide Semiconductor (CMOS) devices have allowed the progressive miniaturisation of these Micro Electro Mechanical Systems (MEMS) [1]. These advances have been adapted by and contributed to 'on-chip' tools used in the biotechnology area and, as a consequence of these advancements, miniaturised and self contained systems known as Micro Total Analysis Systems (µTASs) have been developed. The ability to fabricate micron scale two dimensional arrays of sensors on silicon or glass substrates became possible in 1990's

and led to the invention of the deoxyribonucleic acid (DNA) microarray. This approach was translated across many biomedical applications including protein, cellular, tissue and antibody arrays. However, microarrays are passive in nature and information is normally collected by external analytical equipment. The term Lab-On-a-Chip (LOAC) evolved to describe systems where increased functionality was integrated onto a chip. LOAC devices can integrate one or more laboratory functions onto plastic, glass or a silicon chip and are already commonly used in chemical analysis, environmental monitoring and biomedical diagnostics [2].

However, it is the biomedical application of these LOAC devices that may yield unsurpassed results when used in the analysis and manipulation of biological material. The development of such devices could greatly reduce the skill levels required to obtain complicated bio analysis and reduce the cost of such analysis by reducing the amount of chemicals that tend to be required in any biomedical test. Micro-fabrication will also allow the reproduction of the exact specification of a particular LOAC type and hence increase the accuracy and repeatability of any one specific diagnostic test.

It is worth noting that the recent development of electronic media devices, especially the variants in cell phone technology, lends itself extremely well to the LOAC utilisation. It is feasible that the new generation of portable devices would be capable of running application software that would drive a LOAC to analyse the required biological material. The resulting analytical data could then be transmitted to a medical database or to a medical expert for further analysis. This would then allow valuable resources, doctors and nurses for example, to be used far more effectively in that a simple analysis would filter out cases that do not need immediate medical attention or at least allow the correct prioritisation.

Examples of this are that most cell phones are already capable of taking pulse readings, by using either the camera or microphone components of the phone itself, and have the application software to record and plot blood sugar levels but unfortunately lack the integrated tests that a LOAC device would provide to make the test genuinely self contained [3].

Traditional chemical and biological analysis requires highly skilled technicians working in centralised laboratories. These laboratories contain expensive specialised equipment that is large and expensive in nature. The modern trend is to simplify this analysis so that an untrained person can effectively perform the required analysis; some examples of this are blood glucose tests used for diabetic patients and pregnancy tests that can be purchased in any high street chemist. The LOAC can expand this trend of simplification by offering a simple, reliable and portable laboratory with the potential of working with battery operated analytical equipment [4]. This not only offers modern convenience but can be essential when working in remote field conditions. This is particularly important in countries with poor healthcare resources where the rapid diagnosis of infectious diseases is extremely important, as these countries may well have the drugs to cure an illness but not the diagnostic expertise to identify the cause and then administer the applicable drugs [5].

However, it is important to remember that a LOAC working as part of a much larger static integrated analysis system, offering reliable and consistent results, is also an important LOAC characteristic.

LOAC's also have important applications in the environmental sector where real time monitoring is much more effective than traditional sampling; for example the monitoring of treated waste water that is fed back into a fresh water stream is much more effective than measuring on a periodic basis as severe damage can be inflicted on this type of eniviroment

in a very short time period. The LOAC device does not necessarily require direct human interaction and therefore is even more useful in harsher climates.

Additionally, the points of measurement in environmental applications can be remote and may require solar or wind generation to power the system, the low power requirement of CMOS devices then lends itself perfectly to the LOAC application. There are another two important advantages in using CMOS technology for LOAC applications. Firstly, the signal in a CMOS device is conditioned by means of dedicated circuitry increasing the signal quality. The second advantage is that large numbers of electrodes and transducers can be integrated onto the chip. The use of CMOS therefore yields large numbers of electrodes on a signal chip with excellent signal quality giving good signal-to–noise ratios [6]. The functionality of these LOAC devices can be divided into three categories: sensors, actuators and readout circuits [7]. As a sensor a LOAC can be used to examine the optical, magnetic, electrical or thermal properties of the target sample [8]. The actuator application is used to apply electrical, electromagnetic or mechanical forces on a sample. The readout circuit application is used to interpret and manipulate the output signal of the sample and then interface to a computer or piece of test equipment.

This thesis will concentrate on the sensor functionality of a LOAC and in particular optical imaging, which is one of the most powerful, non intrusive method of detection used in biomedical and medical applications. The optical structure to be researched in this program is intended to be integrated onto a cell positioning structure of a LOAC platform capable of cell separation and manipulation. The optical structure will extend from the top to bottom surface of the LOAC and will be compatible with CMOS Integrated Circuit (IC) technologies. The CMOS technology provides the potential of high cell count sorting and positioning whilst the new structure will allow the ability to analyse the cells *in situ* using optical techniques

through the substrate leaving the top surface available for other techniques such as Atomic Force Microscopy (AFM). The dilemma with optical imaging is that it does not offer itself easily to the type of miniaturisation that is typically involved in MEMS devices due to the fact that optical measurement equipment is made up of bulky components such as light sources and microscopes. The typical LOAC device will rely on charge-coupled devices (CCD) which will always require the information to be interfaced to another piece of diagnostic equipment. However, if the target specimen could be tested by the use of an adapted LOAC device that uses optically transparent windows throughout the structure in conjunction with the new Biotechnology analytical techniques, such as Epi-fluorescence microscopy[9], there would be huge advantages in cost, robustness and accuracy. Epi-fluorescence microscopy is a method of fluorescence microscopy that is used in the analysis of biological samples. An excitation light is passed through the microscope objective lens and then onto the specimen as shown in Figure 1[9].

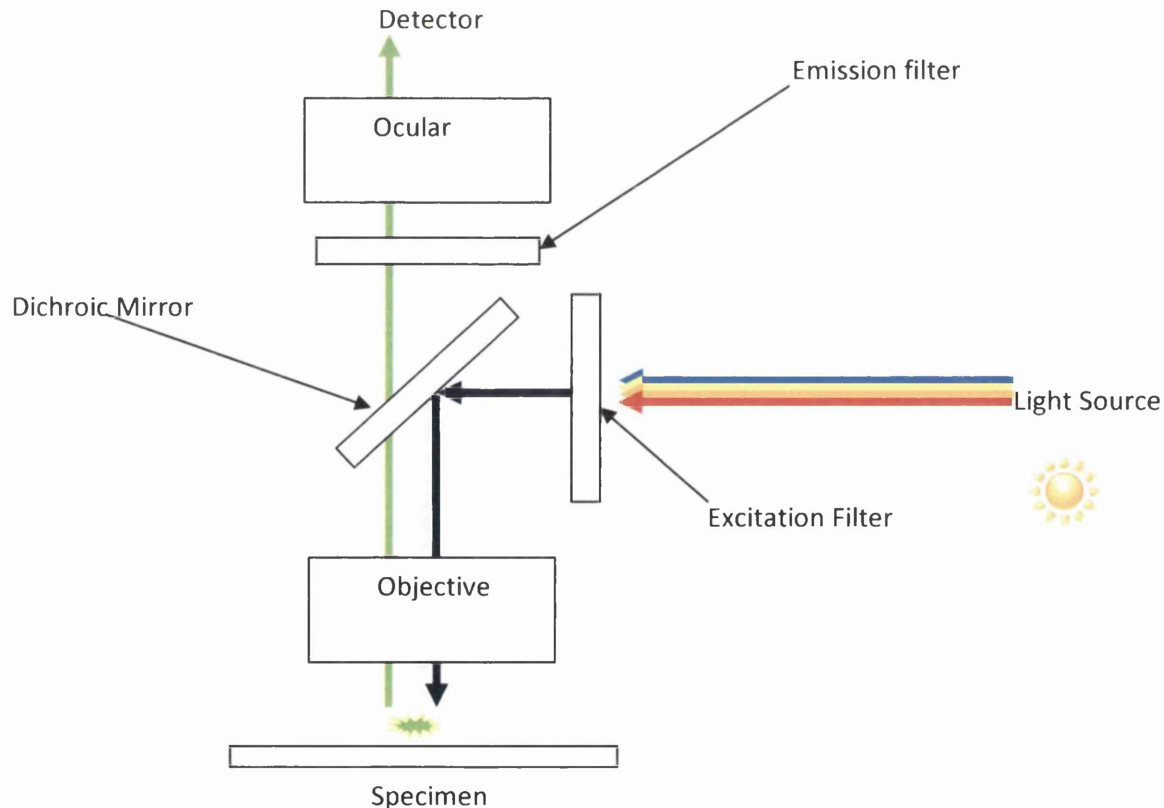


Figure 1 Schematic of a fluorescence microscope

A fluorescence microscope is an excellent example of a larger more expensive piece of equipment that could be integrated on a cheaper LOAC device that would give consistent reliable results with a large throughput. The effect of CMOS devices providing complex low cost digital devices is very apparent in modern electronic equipment. Almost any function can be implemented into an integrated circuit if the potential economic award justifies the investment for the circuit design, the consequent manufacturing process development and the purchase of the required capital equipment [10]. However, it is highly likely that such a LOAC device would be used in third world countries where there is little available money for expensive analytical programs. Therefore keeping manufacturing costs to a minimum would be a fundamental contributor to the uptake of such a device, not to mention the margins and the subsequent profitability of any LOAC used in non-medical applications would be greatly improved. So there are obvious advantages in designing a LOAC device that can be manufactured using existing processing technologies, in other words trying to ensure that the only development costs would be the LOAC layout, control circuitry design and device simulation. The cost of manufacture can be further improved by keeping the number of manufacturing process steps that are used to a minimum, ensuring where possible that the materials used are cheap and uncomplicated to deposit and manipulate into the desired structure. Clearly there is a balance to be determined between the device functionality and performance versus the cost of the device fabrication.

Finally the power consumption levels of such a device would need to be kept as low as possible as it is likely that device would be used in remote locations that do not have reliable nor abundant sources of energy and as a consequence the device would need to be capable of running of low power battery packs[11].

The contents of this thesis are as follows:-

- Chapter 2 describes design objectives the methodology and the fabrication steps that was used in creating the optical structure.
- Chapter 3 shows the design of experiments and corresponding analytical techniques that was used to determine the optimum material and dimensions of the optical window component to be used in the new structure.
- Chapter 4 shows the design of experiments and corresponding analytical techniques that was used to determine the optimum dimensions of the optical via component to be used in the new structure.
- Chapter 5 shows the results of the optical transmission tests of the completed optical structure.
- Finally in Chapter 6 conclusions are drawn and a produced scope of a future program of work is presented.

2. Methodology for the Manufacture of the Structure

2.1. Objectives

The objective is to develop an optical window that can be used in a LOAC structure that will allow the use of suitable analytical equipment, such as Epi-fluorescence. The optical window must possess the optical characteristics to enable any excitation light to pass without any serious attenuation, that could prevent the microscope objective allowing the analysis of the biological specimen, and possess the mechanical strength required to keep the LOAC integral structure intact. This would consist of an Optical Via and an Optical Window as shown in Figure 2.

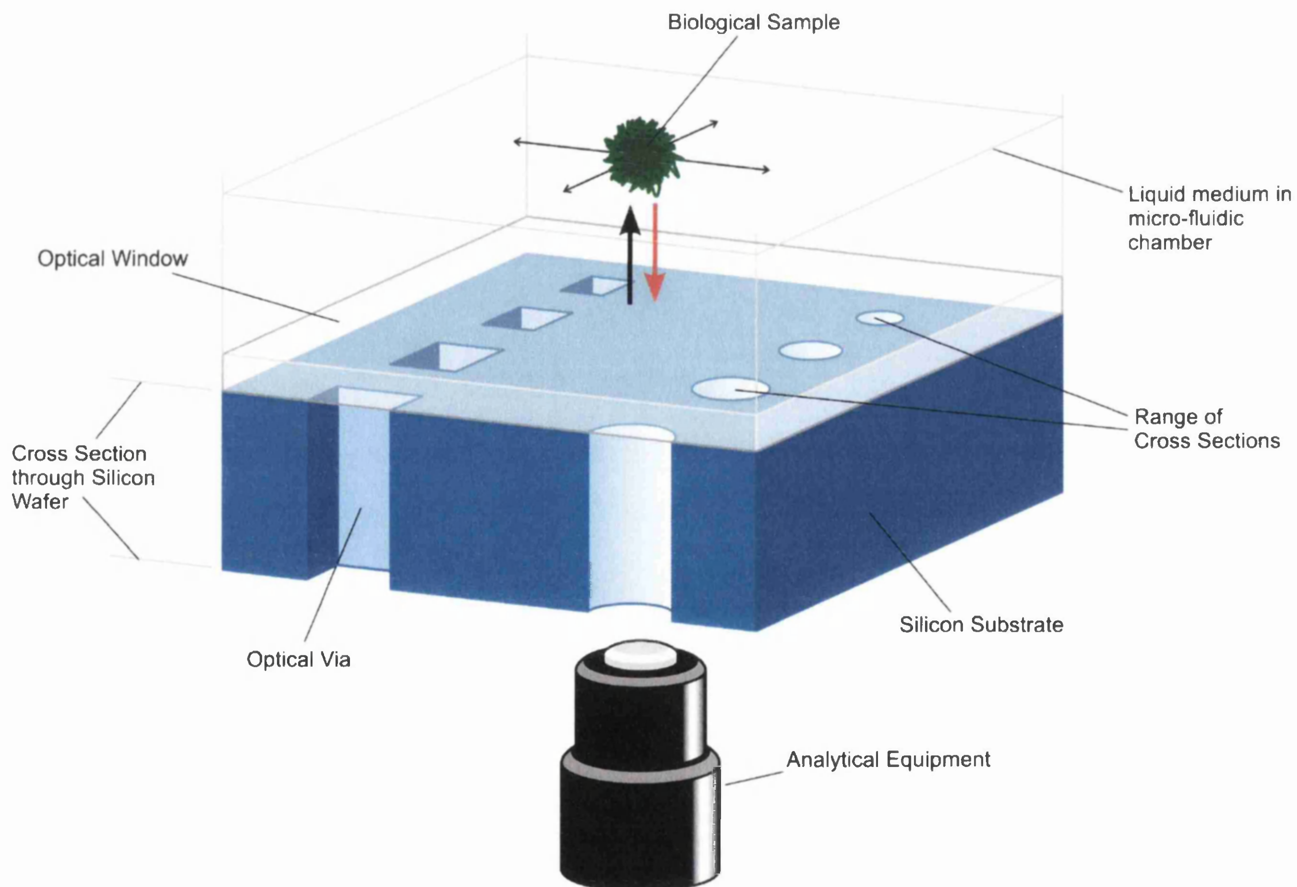


Figure 2 Optical Via and Window

It would be advantageous if any biological sample could be applied directly to the optical window and therefore negating the need for another protective layer, this would not only improve the transmission properties of optical window but also save on manufacturing costs.

Since any biological sample would be extremely prone to any form of contamination then the window will need to be sterile. The sterilisation could form part of the manufacturing process of the device but realistically any such sterilisation would need to be protected by a form of vacuum packaging again adding to costs. The best method of sterilisation would be at the time when the LOAC is about to be used, by soaking the window in a solution, for example Ethanol or Hydrogen Peroxide, for a period of time immediately prior to use. Also if the surface of the optical film is reasonably uniform then the transmission properties will be improved and this may prevent the need of any anti reflecting coating to be applied.

The optical structure will consist of two parts, the optical window and the optical via in which the window is seated, as shown in 2. The optical window will consist of a silicon based film. The via will be formed by etching through a silicon substrate that is 100mm in diameter, this type of substrate is referred to in the semi-conductor industry simply as a wafer. The standard thickness of a 100mm silicon wafer is 525µm. To perform a TSV etch on a 525µm wafer may be difficult as the profile of the etch may degrade severely past a depth of 400µm. However, the thinner wafers that are 400µm in thickness are expensive, difficult for the automation robots used in the semiconductor industry to handle and by their nature of their thickness inherently brittle. It is for this reason that the experiments will be divided into etching wafers of a thickness of 400µm and 525µm, in the hope that the thicker 525µm wafer can be etched successfully.

The Optical Window must possess the optical properties that will allow the operation of the transmission spectrum of optical analytical equipment for example Epi-fluorescence

microscopy uses a spectrum which is typically in the range of 400nm to 1000nm. If it is assumed that excitation light emitted from the microscope is collimated, i.e. light whose rays are nearly parallel, then the excitation light will lose intensity as it is reflected, absorbed or scattered by the optical window. Additionally the window should have a reasonable level of self planarization and the mechanical strength to form part of the structure of the LOAC device.

The mechanical strength of the film will be influenced by the thickness of the film but the thicker the film the more likely that the inherent stress will start to bow the wafer and cause integration issues e.g. the alignment of dual masks becomes extremely difficult on an over stressed wafer.

Additionally there is a requirement to be able to assess the optical transmission properties of the optical window to try and determine the suitability for a range of applications as well as trying to test the suitability of biomedical optical applications which is typically in the range of 400nm to 1000nm. In other words two structures will need to be manufactured the first a smaller and therefore stronger structure to be able to hold and analyse the biomedical matter and a larger structure which will large enough to allow the fibre optics of the emission spectroscopy analytical equipment to be inserted into the window and thus stop any interference caused be the walls of a smaller optical via.

2.2. Manufacturing Process flow of the Structure

2.2.1. Substrate Selection

Since the beginning of the semiconductor industry, silicon has been the most widely used substrate material. Silicon is very low cost, exhibits much lower leakage currents than other substrates, such as germanium, and is easily doped with n-type or p-type, essential for IC devices. Mechanically, silicon has a Young's Modulus of 190GPa which is similar to that of steel (210GPa). Since silicon is a light material with a density of 2330Kg/m^3 which is lower than that of aluminium (2700kg/m^3) it has a very high strength to weight ratio. It is for these reasons that silicon is also used for MEMS, optoelectronic subsystems and micro fluid applications [12]. Interestingly our application will encompass all three applications and therefore makes silicon the material of choice. The substrate size of 100mm was selected for cost reasons more than scientific ones in that they are cheaper. This is the smallest size in mainstream MEMS manufacture and it is a simple matter to mount the 100mm wafers to a larger size substrate. Indeed the substrates were run on tools set at 100mm, 150mm, 200mm and 300mm.

If 100mm processing systems were not available, as in the case of the etch processing, the substrate was bonded to the larger substrate using a mounting adhesive. This adhesive exhibits high bond strength and adheres readily to metals, glass and ceramics. When processing was complete, the adhesive was removed by reheating and cleaning the substrate with acetone. When the 100mm substrates were run through the chemical vapour deposition (CVD) systems they were simply placed on the larger 300mm substrates. The photolithography equipment used 100mm settings as extreme levels of accuracy are required and mounting on a larger substrate would have caused alignment issues.

2.2.2. Step 1- The application of the silicon based layer to form the Optical Window.

To manufacture the optical window of the structure a silicon based film is required to be deposited on the backside of the wafer. The film was deposited on the backside of the wafer as the front side on the wafer tends to be highly polished and therefore more suitable for imprinting the pattern of the design of the structure using photolithography equipment.

The film was deposited using CVD equipment. CVD allows deposition to take place at relatively low temperatures and offers a wide range of accurately controllable composition and layer structures that are often difficult or impossible to obtain using other techniques. CVD can be defined as a material synthesis method in which the constituents of the vapour phase react to form a solid film at some surface [13].

There are three main methods of depositing silicon films within the semiconductor industry Atmospheric Pressure Chemical Vapour Deposition (APCVD), Low Pressure Chemical Vapour Deposition (LPCVD) and Plasma Enhanced Chemical Vapour Deposition (PECVD) [14].

APCVD process chambers were the first to be used in the semiconductor industry. They operate at atmospheric pressures which allow the process chamber design to be relatively simple and also tend to give high film deposition rates. However, APCVD is susceptible to gas-phase reactions, and consequently unwanted particles and the films typically exhibit poor step coverage [14].

LPCVD reactors, although an improvement on the APCVD, still exhibit the same shortcomings and have the added disadvantage of requiring a high process temperature [14].

The advantage of a PECVD process is that lower deposition temperatures can be used. However, the lower temperature of the PECVD process can have a negative effect on the quality of the film being deposited [15]. Oxides deposited at lower temperatures contain more Silanol and water impurities and tend to be more porous than those deposited at higher temperature. However by adjusting the process parameters of the PECVD the Silanol concentration and water contamination can be reduced and allow superior film qualities to be exhibited.

A Plasma can be defined as a partially ionised gas with equal numbers of positive and negative charges [16]. In the process chamber, the plasma is generated between two parallel plate electrodes with radio frequency (RF) power applied to them. The bottom electrode is what the substrate sits on and is commonly referred to as the platen, and the upper electrode through which the process gases are introduced, is known as a showerhead. When RF power is applied to the showerhead, the platen is set to earth potential, generating an electric field between the two electrodes. Three important processes will then take place known as ionisation, excitation-relaxation and dissociation [17].

Electrons are accelerated by the field and gain energy. The energetic electrons collide with the atoms and molecules inside the chamber and cause ionisation which generates more electrons. Very soon the whole chamber fills with electrons and ions and the plasma is generated and stabilised. Excitation-relaxation collisions in the plasma are what cause plasmas to have coloured glows. A species is excited by an electron and then upon relaxation, light of a particular wavelength is emitted. Oxygen glows are grey blue, nitrogen is pink and fluorine red. In addition to the excitation-relaxation collisions an electron can collide with a species and generate free radicals which are molecular fragments with unpaired electrons

(this is called dissociation). These free radicals are chemically very reactive and therefore will dramatically increase the reaction rate for both CVD and for that matter Etch processes.

Plasma enhanced CVD requires the control and optimisation of RF power density and frequency in addition to those conditions important in an LPCVD process such as gas composition, flow rate, deposition temperature and total pressure. Like the LPCVD process, at low temperatures, the PECVD process is surface reaction limited so good substrate temperature control is necessary to ensure film thickness uniformity.

In order to explore the type and thickness of silicon films that could be deemed suitable for use as the optical window PECVD methods will be used to deposit the following range of films:

Silicon Dioxide (SiO_2)

SiO_2 consists of flexible and adjustable Si-O-Si bridge bonds and used mainly as an inter-layer dielectric to electrically isolated conductive layers of the integrated circuit from each other [18]. It is also used as a mask or capping layer, but more importantly can be used as a passivation layer. It is prudent to try and use this simple film from a cost and integration perspective. In this application it may be that strength is more important than hardness and its low stress levels may be able to make it suitable as it will not tend to crack and then peel away from the optical via when thicker layers are deposited.

However, Silicon Dioxide can display poor "Gap Fill" properties on smaller structures and is not very conformal and may require a Chemical Mechanical Polish (CMP) to make this film a practical solution [19]. CMP is a method of mechanical polishing the film surface by using a rotating abrasive pad. This causes the high spots on the surface of the film to be polished

before the low spots. The chemical component is given by a slurry that acts as a lubricant and helps to break down the SiO_2 bonds at the surface of the film [20].

Silicon Nitride (Si_3N_4)

Si_3N_4, is a hard, dense, refractory material. Its structure is quite different from that of silicon dioxide: instead of flexible, adjustable Si-O-Si bridge bonds, the Si-N-Si structure is rigid and the material is much more constrained in structure than that of silicon dioxide [21]. As a consequence, silicon nitride is harder, has higher stress levels, and cracks more readily. This harder layer may be suitable but there is a possibility that it will crack and then peel away from the optical trench. Silicon nitride also has poor gap fill properties and is also not very conformal.

Silicon Oxynitride (SiO_xN_y)

SiO_xN_y films combine some of the useful physical characteristics of SiO_2 and Si_3N_4 and are consequently used extensively in the MEMS industry. Silicon oxynitride contains significant amounts of excess silicon and hence the mechanical stress in the Silicon Oxide Nitride is lower than that of other oxides [22]. It has excellent thickness uniformity and is very conformal [23]. For optical devices is it particularly attractive as it has high transparency levels over a wide wavelength range [24].

Tetraethylorthosilicate (TEOS)

TEOS has excellent gap fill properties and is very conformal [25]. It also has the advantage of being deposited on substrate at much lower temperatures than other PECVD oxide films and as such will not tend to damage any metal layer in close proximity. It also has excellent mechanical strength which is essential for this application [26]. However, it can be a poor moisture barrier and may require further encapsulation to make the film practical for this

application [27]. So this film will only be considered if the other films are not deemed suitable.

The thicknesses and types of films listed in table 1 were deposited as an initial design of experiments to determine a suitable film to be used for the optical window.

Wafer Number	Target Thickness (μm)	Film Type
1-5	1.5,2,3,4 & 5	Silicon Dioxide (SiO_2) at 350°C
6-10	1.5,2,3,4 & 5	Tetraethylorthosilicate (TEOS) at 175°C
11-15	1.5,2,3,4 & 5	Tetraethylorthosilicate (TEOS) at 350°C
16-20	1.5,2,3,4 & 5	Silicon Oxide Nitride (SiO_xN_y) at 175°C
21-25	1.5,2,3,4 & 5	Silicon Nitride (Si_3N_4) at 300°C

Table 1 Silicon films deposited to form the optical window

These ranges of thicknesses were chosen as they were deemed the best compromise between optical properties, stress, non uniformity and mechanical strength. The basis of this question is a simple one - can a film be deposited thin enough to be of use with optical analytical equipment and be strong enough to remain intact? This question can be further extended in that could the film also have enough moisture barrier properties so that no further manufacturing steps are required to allow direct contact of the window with the bio material? Additionally, it was unknown whether the optical window needs to be protected so wafers were run with and without protection. The reason for this assumption is that the etch required is a TSV etch which would require the silicon film to placed face down onto the electrostatic