

A

Project Report

On

“Optimization of process parameter for the Deposition of SiO₂ thin film using Thermal CVD.”

SUBMITTED TO

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In the Academic year

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CERTIFICATE

This is to certify that the project report entitled

“Optimization of process parameter for the Deposition of SiO₂ thin film using Thermal CVD.”

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This is to certify that, this is the original independent work of the student himself, under my super vision during his stay in this University in the 2022-2023 for the partial fulfillment of project report. To the best of my knowledge all the references studied for the preparation in this work have been properly acknowledged.

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(Mr. Patil Manish Gokul)

Abstract

The importance of an integrated approach involving synthetic chemistry, physical chemistry, and chemical engineering to the development of thermal chemical vapor deposition (TCVD) processes for the deposition of thin-film, electronic materials. Initially we refurbished the old TCVD system and updated the new Quartz tube, renewed the internal wiring, DTC (Digital Temperature controller) on it to control the high and low temperature for deposition of uniform and smooth thin films. And as result the system is successfully working today. for the deposition SiO₂ thin films on the system through understanding and optimization of the following process parameters such as I)Substrate temperature, II)TEOS bubbler temperature ,III)Oxygen (O₂) flow rate, IV)TEOS flow rate, V)Process time are important accordingly, the optimization of process parameter is being done these days. Thereafter, chemical and structural characterization will be conducted by Ellipsometry.

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Chapter 1

Introduction

Introduction

The development of thermal chemical vapor deposition (TCVD) processes for the deposition of thin-film electronic materials necessitates an integrated approach that combines the expertise of synthetic chemistry, physical chemistry, and chemical engineering. This project report highlights the significance of this integrated approach in optimizing the TCVD process for depositing SiO_2 thin films. By refurbishing an old TCVD system, updating critical components, and optimizing process parameters, we have successfully achieved a functional system capable of depositing uniform and smooth thin films.

The integration of synthetic chemistry, physical chemistry, and chemical engineering is essential for the success of TCVD processes. Synthetic chemistry allows us to design and synthesize suitable precursor materials with controlled properties. These precursors play a crucial role in achieving the desired characteristics of the thin films. Physical chemistry provides insights into the underlying thermodynamics and kinetics of the deposition process, enabling the optimization of process parameters. Chemical engineering facilitates the practical implementation of these processes by designing and modifying the TCVD system to control temperature, flow rates, and deposition conditions.

The initial phase of this project involved refurbishing the old TCVD system to ensure its proper functioning. Upgrading the Quartz tube, renewing the internal wiring, and implementing a Digital Temperature Controller (DTC) were crucial steps. The new Quartz tube provides a clean deposition environment, while the renewed wiring ensures consistent power supply. The DTC allows for precise control of high and low temperatures, facilitating the deposition of uniform and smooth thin films.

Optimization of process parameters is a key focus of this project. Several parameters have been identified as crucial for achieving optimal deposition results. The substrate temperature plays a significant role in controlling the growth kinetics, crystallinity, and surface morphology of the deposited thin films. By carefully adjusting the substrate temperature, we can achieve the desired film properties. The TEOS bubbler temperature is another important parameter as it influences the vaporization rate and stability of the precursor. Precise control of the TEOS bubbler temperature ensures a consistent supply of TEOS, which is crucial for controlled film growth.

The oxygen (O_2) flow rate regulates the oxidative environment during deposition and influences the film's density, and composition. Optimization of the O_2 flow rate allows us to achieve the desired film properties. Similarly, the TEOS flow rate is essential in determining the availability of precursor molecules for film growth. By optimizing the TEOS flow rate, we can ensure a sufficient precursor concentration and deposition rate, enabling the deposition of films with the desired thickness. The process time determines the duration of the deposition process, impacting film thickness and other structural properties. Optimizing the process time allows us to achieve consistent and reproducible results.

Following the optimization of the TCVD process parameters, thorough chemical and structural characterization of the deposited thin films will be conducted. Techniques such as Ellipsometry will be employed for this purpose. Ellipsometry provides valuable information about film thickness and refractive index, indicating film quality and uniformity.

1.2. Background

Thin films refer to layers or coatings of material that have a thickness ranging from a few nanometres to a few micrometres. These films can be deposited onto a substrate using various techniques and find applications in numerous fields, including electronics, optics, energy, coatings, and more. Understanding the background of thin films involves exploring their properties, fabrication methods, and key scientific principles.

1.2.1. Properties of Thin Films:

Thickness: Thin films have dimensions on the nanoscale or microscale, with thicknesses typically ranging from a few atomic layers to a few micrometres.

Interface: Thin films are characterized by the interface between the film and the substrate. This interface often influences the film's properties and performance.

Uniformity: Achieving uniform thickness and composition across the film is essential for desired functionality.

Adhesion: The ability of a thin film to adhere to the substrate is crucial for its stability and longevity.

Optical Properties: Thin films exhibit optical phenomena such as interference, reflection, transmission, and absorption, making them important in the field of optics and photonics.

Electrical Properties: Thin films can exhibit electrical conductivity or insulating behaviour, depending on the material and fabrication techniques used.

1.2.2 Fabrication Techniques:

Physical Vapor Deposition (PVD): PVD methods involve vaporizing the source material and condensing it onto the substrate.

- Vacuum evaporation
- Sputtering
- Molecular beam epitaxy(MBE)

Chemical Vapor Deposition (CVD): CVD involves the reaction of precursor gases on the substrate to deposit a thin film.

- Atmospheric pressure chemical vapor deposition(APCVD)
- Low pressure chemical vapor deposition(LPCVD)
- Plasma Enhanced chemical vapor deposition(PECVD)
- Photochemical vapor deposition(PCVD)
- Thermal Chemical vapor deposition(TCVD)

- Plasma Enhanced Atomic Layer Deposition (PECVD)
- **Sol-Gel Deposition:** This technique involves the conversion of a precursor solution into a solid film through hydrolysis and condensation reactions.
- **Spin Coating:** In spin coating, a liquid solution is spread onto a rotating substrate, creating a thin film as the solvent evaporates.

Through this different technique the main trust of the technology has been the controlled fabrication of thin films, which include control over composition, stoichiometry, crystallinity, density and microstructure.

Chapter 2

Theory of chemical Vapor Deposition

2.1 What is Chemical Vapor Deposition?

Chemical Vapor Deposition (CVD) is a process used to deposit thin films or coatings onto a substrate by introducing reactive gases into a reaction chamber. These gases react or decompose on the substrate surface, resulting in the formation of a solid film. CVD is widely used in various industries, including electronics, optics, and materials science.

2.1.1 Basic aspects of Chemical Vapor Deposition (CVD):

Process: CVD involves the reaction of volatile precursor gases in a reaction chamber to deposit a solid film onto a heated substrate. The precursors are introduced into the chamber, where they undergo chemical reactions or decomposition on the substrate surface. The resulting reaction by-products are usually gaseous and are removed from the chamber.

Reactor: The CVD reactor is designed to provide a controlled environment for the deposition process. It typically consists of a reaction chamber, heating elements, gas delivery systems, and exhaust systems. The reactor can operate at atmospheric pressure, reduced pressure, or even under vacuum conditions, depending on the specific CVD technique.

Substrate: The substrate is the material onto which the thin film is deposited. It can be made of various materials such as silicon, glass, metals, ceramics, or polymers. The substrate's surface properties and preparation play a crucial role in achieving good film adhesion and quality.

Precursor Gases: CVD relies on the use of precursor gases, which are typically volatile compounds or mixtures. These gases contain the elements necessary for film formation. For example, in the deposition of silicon dioxide (SiO_2), a common precursor gas is silane (SiH_4) combined with oxygen (O_2) or nitrous oxide (N_2O).

Energy Source: CVD requires an energy source to drive the chemical reactions and promote film growth. This energy can be provided through various means, including thermal energy, plasma, or light (photolysis). The choice of energy source depends on the specific CVD technique and the desired film properties.

Temperature: The substrate is typically heated during CVD to facilitate the chemical reactions and promote film growth. The temperature range depends on the materials involved and the specific CVD process. In thermal CVD, temperatures are often in the range of 500°C to 1000°C , while in some PECVD or ALD processes, lower temperatures in the range of 100°C to 500°C can be used.

Film Characteristics: The properties of the deposited film can be controlled by adjusting various process parameters, such as precursor concentration, temperature, gas flow rates, and deposition time. These parameters influence the film's thickness, composition, crystallinity, uniformity, adhesion, and other properties.

Indigenously Developed Thermal Chemical Vapor Deposition System (CVD)



Fig: Indigenously Developed Thermal Chemical Vapor Deposition System (CVD)

2.1.2 Thermal CVD: -

This is the most basic form of CVD, where the substrate is heated to a high temperature, typically above 500°C. The reactive gases are introduced into the chamber, and the chemical reactions occur on the heated surface. Thermal CVD is often used for depositing materials like silicon dioxide, silicon nitride, and polysilicon.

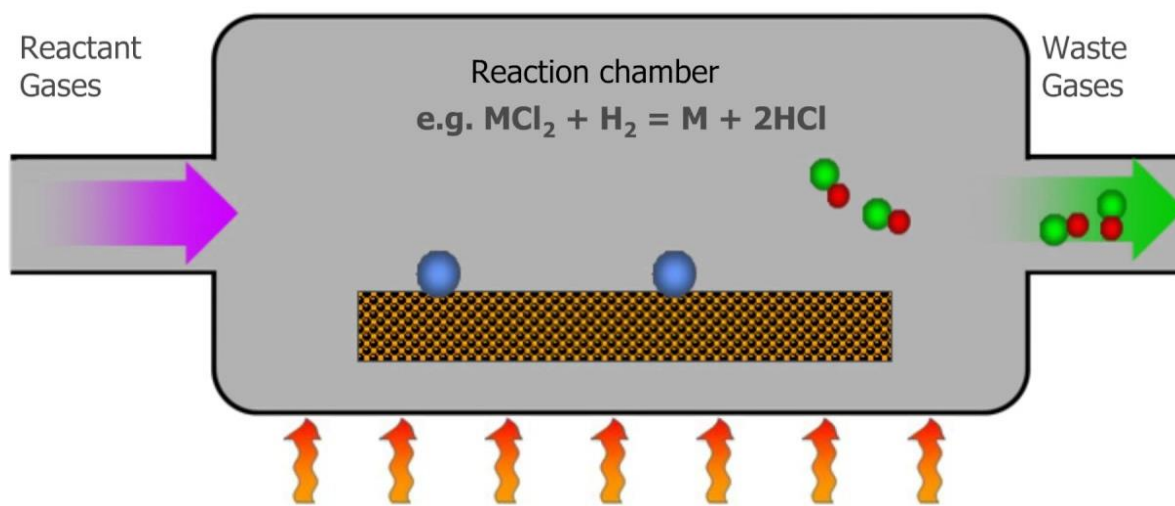


Fig: -Chemical Vapor Deposition (CVD)

2.1.3 Plasma-Enhanced CVD (PECVD): In PECVD, the reactive gases are excited into a plasma state using an electric field or radiofrequency energy. The plasma provides additional energy to the reaction, allowing film deposition at lower temperatures compared to thermal CVD. PECVD is commonly used for depositing silicon-based films and various thin films for microelectronics and photovoltaic applications.

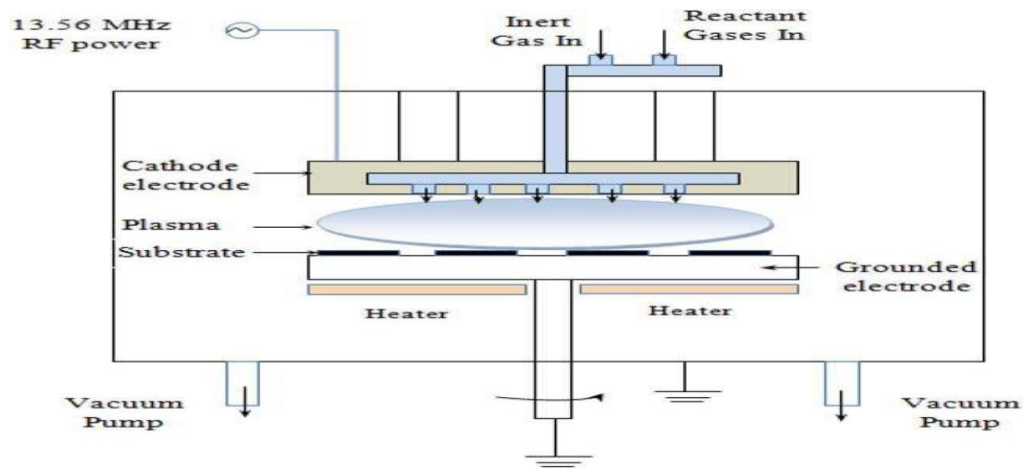


Fig: -Plasma-Enhanced CVD

2.1.4 Low-Pressure CVD (LPCVD): In LPCVD, the reaction chamber is operated at reduced pressures, typically below atmospheric pressure. Lowering the pressure enables better control over the reaction kinetics and results in higher-quality films. LPCVD is frequently employed for depositing materials like silicon nitride, silicon dioxide, and amorphous silicon.

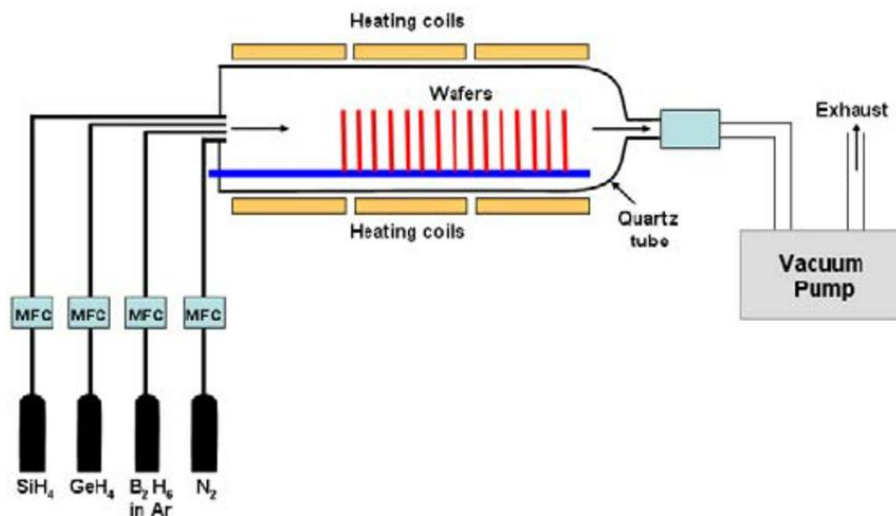


Fig:-Low-Pressure CVD

2.1.5 Metal-Organic CVD (MOCVD): MOCVD is used primarily for depositing thin films of compound semiconductors, such as gallium arsenide (GaAs) or indium gallium nitride (InGaN). It involves the use of volatile metal-organic precursors, which decompose and react on the substrate surface to form the desired compound.

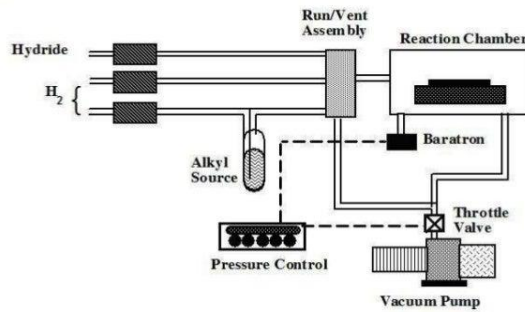


Fig: - Metal-Organic CVD (MOCVD)

2.1.6 Atomic Layer Deposition (ALD): Atomic Layer Deposition (ALD) is a thin film deposition technique used in materials science and nanotechnology. It is a controlled and highly precise method for depositing uniform layers of materials onto a substrate surface with atomic-scale accuracy.

In ALD, the film growth is achieved through a series of self-limiting surface reactions. The process involves alternating exposure of the substrate to two or more precursor gases in a sequential manner. Each precursor reacts with the surface to form a monolayer, and any excess or unreacted precursor is purged before introducing the next precursor. This sequential deposition process allows precise control over film thickness and composition at the atomic level.

ALD offers several advantages over other deposition techniques. Firstly, it enables the deposition of conformal films on complex three-dimensional structures, including high-aspect-ratio features. It also allows precise control over film thickness, even at the sub-nanometer scale. Moreover, ALD can deposit films with excellent uniformity and coverage, making it suitable for applications requiring precise control over material properties.

The versatility of ALD is reflected in its wide range of applications. It is extensively used in the semiconductor industry for fabricating ultra-thin high-k dielectric layers, gate oxides, and metal contacts in advanced electronic devices. ALD is also employed in energy storage and conversion devices, such as batteries and fuel cells, to enhance their performance and stability. Additionally, ALD finds applications in optical coatings, catalysis, protective coatings, and sensors, among others.

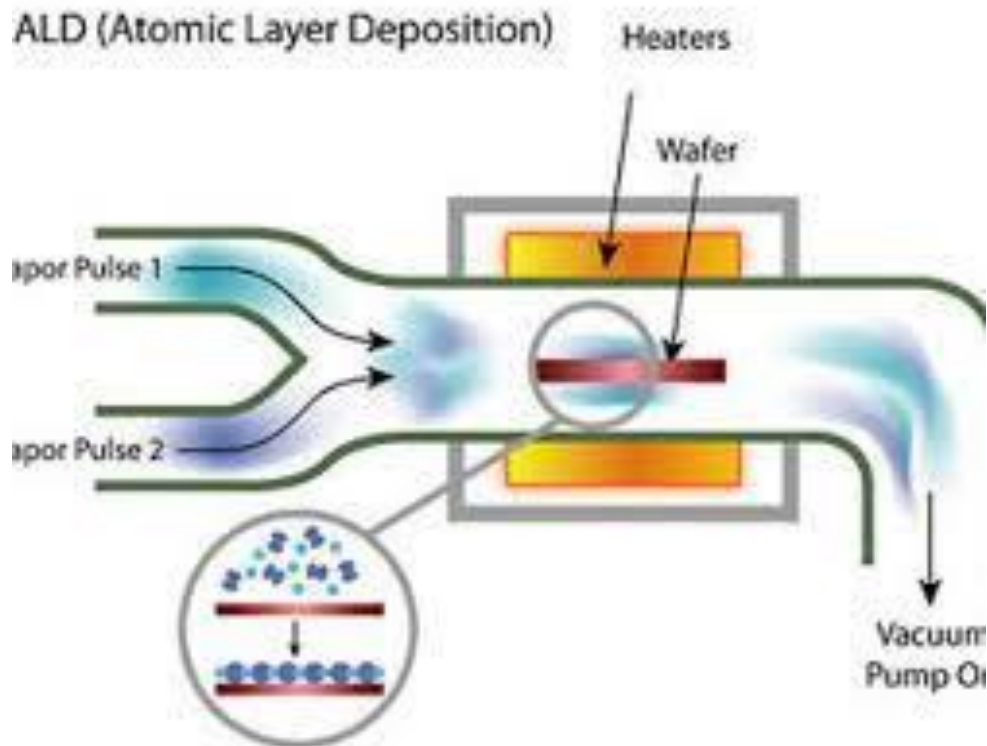


Fig:-Atomic Layer Deposition (ALD)

2.1.7 Plasma-Enhanced Atomic Layer Deposition (PEALD): is a variation of the Atomic Layer Deposition (ALD) technique that incorporates plasma during the film deposition process. PEALD combines the advantages of ALD and plasma processing to enhance film growth and achieve specific material properties.

In PEALD, a plasma source is used to activate the precursor gases, creating a highly reactive environment. The plasma consists of energetic species such as ions, electrons, and radicals, which can facilitate the surface reactions and increase the reactivity of the precursors. The reactive species in the plasma help to break down the precursor molecules, promote surface reactions, and remove byproducts, resulting in enhanced film growth rates compared to conventional ALD.

The use of plasma in PEALD offers several benefits. It allows for lower process temperatures, enabling deposition on temperature-sensitive substrates. The plasma can improve the step coverage and conformity of the deposited films, ensuring uniformity and coverage even on

complex three-dimensional structures. Additionally, the plasma can introduce additional functionality to the films by incorporating specific elements or modifying the film properties, such as the density, porosity, or refractive index.

PEALD has found applications in various areas, including semiconductor manufacturing, energy storage, optical coatings, and nanoelectronics. It is particularly useful for depositing thin films with precise control over thickness, composition, and properties. Examples of materials commonly deposited using PEALD include high-k dielectrics, metal oxides, nitrides, and silicon-based films.

By leveraging the benefits of both ALD and plasma processing, Plasma-Enhanced Atomic Layer Deposition provides a versatile and controlled approach for thin film deposition, enabling the fabrication of advanced materials and devices with tailored properties and high precision.

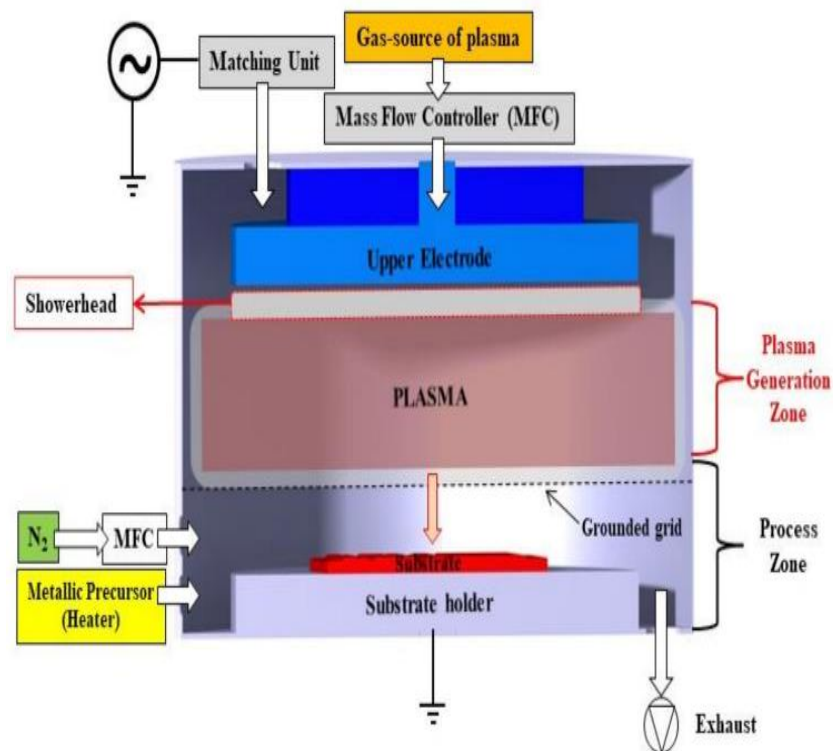


Fig:-Plasma-Enhanced Atomic Layer Deposition (PEALD)

Chapter 3

Schematic Diagram and Components of TCVD

3.1 Schematic Diagram of TCVD:-

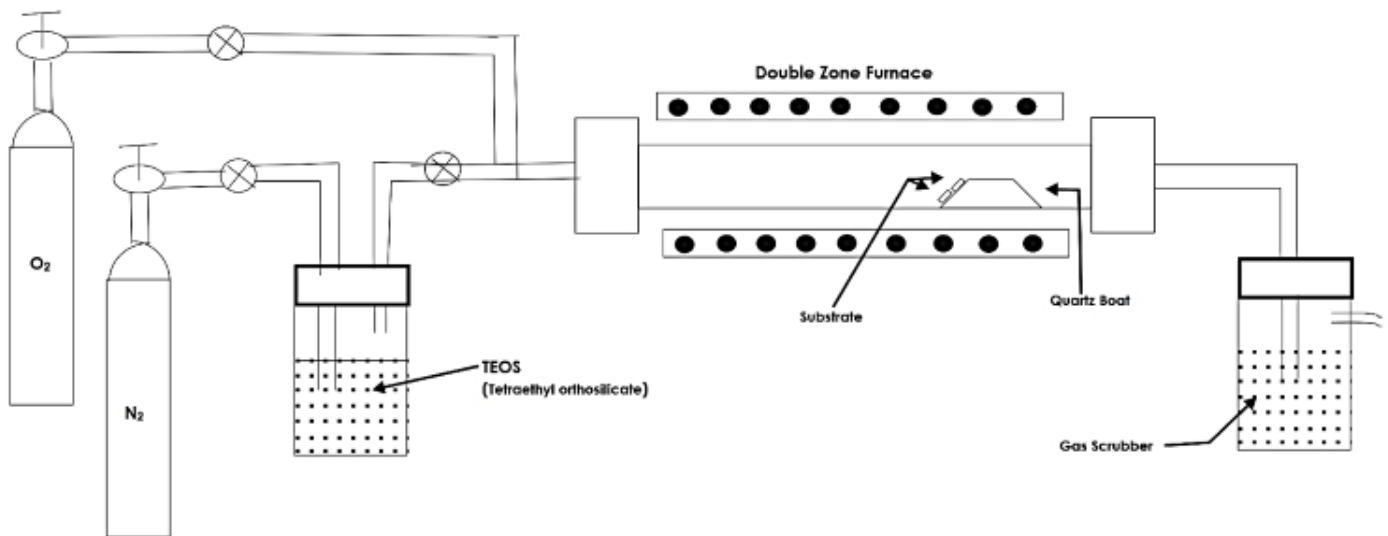


Fig:-Basic schematic diagram of TCVD

3.2 Components of TCVD

The typical components and equipment involved in a CVD system can include: -

- 1) Quartz reactor and boat.
- 2) Single zone furnace with decreasing temperature profile.
- 3) Gas sources and gas line.
- 4) Bubblers.
- 5) Mass Flow Controller (MFC).
- 6) Control panels.
- 7) Digital Temperature Controller (DTC).
- 8) Gas exit module.

3.2.1 Quartz reactor and boat: -

A quartz reactor and boat are components commonly used in various high-temperature processes, including thermal chemical vapour deposition (CVD) and thermal annealing. They are typically made of high-purity quartz due to its excellent thermal stability and resistance to chemical reactions.

A quartz reactor refers to a cylindrical or tubular chamber made of quartz material. It serves as the main vessel where the deposition or annealing process takes place. The reactor is designed to withstand high temperatures required for the specific process and maintain a controlled environment. The quartz material is preferred because it is transparent to the heat source (e.g., infrared radiation) used for heating and allows for good thermal insulation.

Inside the quartz reactor, a quartz boat is often employed as a sample holder or substrate carrier. The boat is shaped like a long, narrow tray or trough, also made of high-purity quartz. It is designed to accommodate the substrates or samples that will undergo the deposition or annealing process. The boat can hold multiple substrates in a controlled arrangement, ensuring uniform exposure to the process conditions.

The quartz boat serves several important purposes. It provides mechanical support and stability to the substrates during the high-temperature process. It also acts as a barrier, preventing direct contact between the samples and the reactor walls, which could lead to

contamination or undesired reactions. Additionally, the boat allows efficient gas flow around the substrates, facilitating uniform deposition or heat treatment.

The selection of quartz for both the reactor and boat is driven by its desirable properties for high-temperature applications. Quartz exhibits excellent thermal shock resistance, high melting point, low thermal expansion, and good chemical inertness. These properties make it suitable for maintaining a stable and controlled environment inside the reactor and ensuring the integrity of the substrate during the process.



Fig: - Quartz reactor and boat.

3.2.2 Single zone furnace with decreasing temperature profile:-

Single zone furnace with a decreasing temperature profile refers to a heat treatment system that consists of a single heating zone but is designed to achieve a gradual decrease in temperature over time. In this setup, the entire furnace operates at a uniform temperature, but the temperature is gradually reduced as the process progresses. The decreasing temperature profile in a single zone furnace can be achieved through various methods, such as adjusting the power input, changing the gas flow rate, or employing cooling mechanisms. The goal is to create a controlled thermal environment where the temperature decreases gradually over a specified duration. This type of temperature profile can be utilized in different heat treatment processes, such as annealing, stress relief, or controlled cooling. The gradual temperature

decrease helps to minimize thermal stress, enhance material properties, or achieve specific microstructural changes.



Fig:-Single Zone Furnance with heater coil assembly



Fig:Single Zone Furnance With Heater coil assmbly and Pack with Silica wool

3.2.3 Gas sources & Gas Lines :-

Gas Source:

The gas source in CVD refers to the container or system that holds the precursor gases required for the deposition process. The choice of gas source depends on the specific materials being deposited. Typically, these precursor gases are in a highly pure form to ensure quality film deposition. Some common examples of gas sources used in CVD include:

Gas cylinders: Highly pure gases, such as Oxygen(O_2) and Nitrogen (N_2), can be stored in compressed gas cylinders. These cylinders are usually equipped with regulators and valves to control the flow of gases.



Fig:- Gas sources, gas line and bubblers

3.2.4 Bubblers: Bubblers are devices used to introduce liquid precursors into the deposition process. Liquid precursors are commonly used in certain deposition techniques, such as CVD. The liquid precursor is placed inside the bubbler, which is typically a glass container with an inlet and outlet for gas flow. The bubbler is heated to a specific temperature, causing the liquid precursor to evaporate and form a vapor. The vapor is then carried by a carrier gas into the reaction chamber, where it participates in the deposition process.

The bubblers serve a vital role in controlling the flow rate and concentration of the liquid precursor. By adjusting the temperature of the bubbler or the carrier gas flow rate, the evaporation rate and subsequently the concentration of the precursor in the gas stream can be controlled. This allows for precise control over the deposition process, ensuring the desired film thickness and composition.

3.2.5 Mass Flow Controller (MFC):-

MFC stands for Mass Flow Controller. It is a device commonly used in various industries, including semiconductor manufacturing and CVD, to precisely control the flow rate of gases.

The Mass Flow Controller consists of three main components:

Flow Sensor: The flow sensor measures the actual flow rate of the gas passing through the MFC. It typically uses thermal, pressure, or differential pressure-based principles to determine the flow rate accurately.

Control Valve: The control valve is responsible for adjusting the flow rate of the gas based on the desired setpoint. It modulates the gas flow by opening or closing to allow more or less gas to pass through.

Electronics and Control Circuitry: The electronics and control circuitry receive feedback from the flow sensor and compare it to the setpoint value. They regulate the control valve's position and speed to maintain a constant flow rate, compensating for any variations or disturbances.

The MFC is often integrated into the gas line in CVD systems or other processes where precise control of gas flow is critical. The desired flow rate is typically set through the control interface of the MFC, either manually or through a computerized system.

MFCs offer several advantages, such as:

Accurate and Repeatable Flow Control: MFCs can provide precise and stable gas flow rates, allowing for consistent and reproducible process conditions.

Wide Range of Flow Rates: MFCs are available in various flow rate ranges, from a few sccm (standard cubic centimeters per minute) to several slm (standard liters per minute) or more, accommodating different process requirements.

Gas Composition Flexibility: MFCs can handle a wide range of gases, including corrosive,

reactive, and specialty gases, making them versatile for different applications.

Real-Time Monitoring and Control: MFCs often provide real-time feedback on the actual flow rate, allowing for continuous monitoring and adjustment of the gas flow during the process.



Fig:-Mass Flow Controller (MFC)

3.2.6 Control Panel:-

In a chemical vapor deposition (CVD) system, the control panel serves as the central interface for monitoring and controlling various parameters and components involved in the deposition process. It allows operators to set up and adjust process conditions, monitor critical parameters, and ensure the system operates safely and efficiently. The specific layout and features of the control panel may vary depending on the CVD system manufacturer and configuration, but here are some common elements you might find on a CVD control panel:

Temperature Control: CVD processes often require precise temperature control. The control panel provides temperature settings for the deposition chamber, substrate heater, and other temperature-sensitive components. It may display real-time temperature readings and allow adjustment of setpoints.

Gas Flow Control: The control panel typically includes controls for regulating gas flow rates. It allows operators to set the desired flow rates of various precursor gases or carrier gases used in the deposition process. This control may involve individual flow control knobs or digital inputs for mass flow controllers (MFCs).

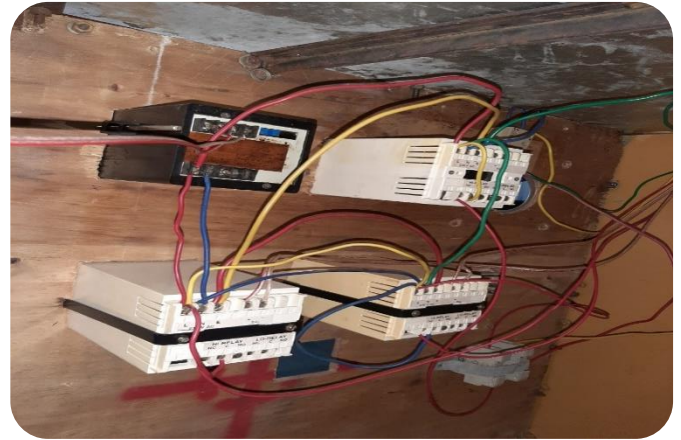


Fig:-Control Panel and Internal Wiring of TCVD

3.2.7 Digital Temperature controller(DTC) :-

A digital temperature controller is an electronic device used to monitor and control temperature in various applications. It is designed to provide accurate temperature regulation and stability, making it useful in industrial processes, scientific research, and home automation systems.

Here's how a typical digital temperature controller works:

Temperature Sensing: The controller uses a temperature sensor, such as a thermocouple or a resistance temperature detector (RTD), to measure the current temperature. The sensor converts the temperature into an electrical signal that the controller can process.

Setpoint Input: The user sets the desired temperature using the controller's interface, typically through a digital display and buttons. This value is called the setpoint.

Comparison and Control: The controller continuously compares the actual temperature (as measured by the sensor) with the setpoint. Based on this comparison, it determines whether to activate a heating or cooling mechanism.

Output Control: The controller's output can be used to control various devices, such as heaters, coolers, fans, or valves. For example, if the actual temperature is below the setpoint, the

controller may activate a heater to increase the temperature. Conversely, if the actual temperature is above the setpoint, it may activate a cooling system.

Feedback Loop: The controller continuously monitors the temperature and adjusts the output accordingly. It maintains a feedback loop to ensure that the temperature remains within the desired range. The controller may employ various control algorithms, such as proportional-integral-derivative (PID) control, to optimize temperature regulation.

Display and Interface: The digital temperature controller usually has a digital display to show the current temperature, setpoint, and other relevant information. The interface allows users to configure settings, adjust setpoints, and access additional features.



Fig:-Digital Temperature Controller (DTC)

3.2.8 Gas exit module:-

In Chemical Vapor Deposition (CVD), a gas exit module is a component of the reactor system used to evacuate or vent the gas byproducts generated during the deposition process. During CVD, a precursor gas is introduced into the reactor chamber, where it reacts with the substrate surface to deposit a thin film. The reaction produces byproducts, such as hydrogen gas, carbon dioxide, or water vapor, which can interfere with the deposition process or contaminate the substrate. To prevent these issues, the gas exit module is used to remove the byproducts from the

reactor chamber and maintain a stable gas flow. The gas exit module typically consists of a gas exhaust port, a vacuum pump, and a system of valves and pipes to control the gas flow. The gas exhaust port is located at the opposite end of the reactor chamber from the precursor gas inlet and is connected to the vacuum pump through a series of pipes. The vacuum pump creates a low-pressure environment that allows the byproducts to be removed from the reactor chamber through the gas exhaust port. The valves and pipes in the gas exit module are used to control the flow of gas and to prevent backflow or cross-contamination between the precursor gas and the byproducts. For example, the gas exit module may include a throttle valve to regulate the gas flow rate and a backflow valve to prevent the precursor gas from entering the gas exhaust system.

Chapter 4

Tetraethyl orthosilicate (TEOS)

4.1 What is Tetraethyl orthosilicate (TEOS)?

Tetraethyl orthosilicate (TEOS) is a chemical compound with the formula $\text{Si}(\text{OC}_2\text{H}_5)_4$. It is a colourless liquid that is widely used as a precursor for silicon dioxide (SiO_2) in various applications.

TEOS is primarily employed in the production of silica-based materials, such as glass, ceramics, and thin films. It is a common source of silicon in the semiconductor industry, where it is used to deposit thin films of silicon dioxide by processes like chemical vapor deposition (CVD) or spin coating. These films are essential for insulation, passivation, and dielectric layers in microelectronics and photovoltaic devices.

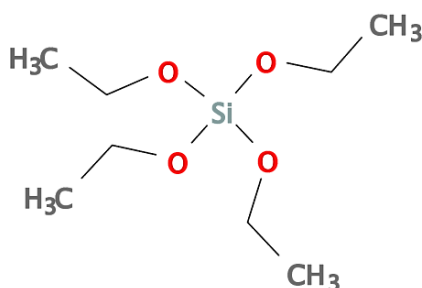


Fig: Chemical structure of TEOS

In addition to its role in silicon-based materials, TEOS also finds applications in other areas:

Sol-gel processes: TEOS is used as a precursor in sol-gel processes, where it undergoes hydrolysis and condensation reactions to form silica-based gels. These gels can be further processed to produce ceramics, coatings, and composites.

Optical coatings: TEOS is utilized in the production of anti-reflective coatings for optical lenses and displays. It helps to improve light transmission by reducing reflections at the surface.

Catalysts: TEOS can serve as a precursor for the synthesis of various catalysts, such as silica-supported metal catalysts used in chemical reactions.

Adhesives and sealants: TEOS is incorporated into adhesives and sealants to enhance their durability, adhesion, and resistance to moisture and chemicals.

It's worth noting that TEOS is highly reactive and volatile, requiring careful handling and storage. It is typically used in well-ventilated environments, and appropriate safety measures should be followed when working with this compound.

4.2 Why we use TEOS as precursor for SiO₂?

Tetraethyl orthosilicate (TEOS) is commonly used as a precursor for silicon dioxide (SiO₂) for several reasons:

Volatility and Reactivity: TEOS is a volatile liquid that readily vaporizes at moderate temperatures, making it suitable for vapor deposition techniques such as chemical vapor deposition (CVD). Its volatility allows for controlled and precise delivery of silicon to the deposition process. Additionally, TEOS undergoes hydrolysis and condensation reactions in the presence of water or moisture, leading to the formation of SiO₂.

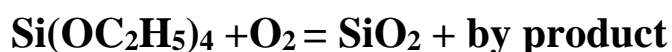
Film Quality and Purity: TEOS-based deposition processes result in high-quality SiO₂ films with excellent uniformity, thickness control, and low defect densities. The controlled vaporization and hydrolysis of TEOS enable the formation of films with desirable properties for various applications, such as microelectronics and optical coatings.

Film Composition Control: TEOS offers the advantage of controlling the stoichiometry of the SiO₂ film. By adjusting the deposition parameters, such as the ratio of TEOS to other reactants or dopants, the composition and properties of the resulting SiO₂ film can be tailored to specific requirements.

Compatibility with Substrates: TEOS-based processes are compatible with a wide range of substrates, including silicon wafers, glass, ceramics, and flexible materials. This versatility makes TEOS a popular choice for SiO₂ deposition in different industries.

Scalability: TEOS deposition processes can be easily scaled up for industrial production. The availability of TEOS in large quantities and its compatibility with high-throughput deposition techniques make it suitable for large-scale manufacturing processes.

➤ **Reaction:-**



Chapter 5

Experimental Setup

5.1 Experimental setup:-

Chemical vapour deposition (CVD) is a fundamental process in the deposition of silicon dioxide (SiO₂), using Tetraethyl orthosilicate (TEOS) thin films for VLSI circuits, MOS devices and optoelectronic devices. We have been using Tetraethyl orthosilicate as a source of Si in addition to O₂ for the deposition of SiO₂ films using thermal-CVD machine.

The reactor is a round quartz tube of 70 cm in length and inner diameter of the tube is 4 cm. Specially designed quartz boat with 5 mm spacing between the adjacent samples is used for holding the substrates inclined in the reactor. The reactor tube is heated and maintained at a constant temperature in the single zone furnace which has the zone length of 10 cm. By keeping the reactor geometry constant, the temperature gradients in the reactor mainly depends on the current flowing through the coil. The hollow stainless steel rings with water-cooling arrangement used to seal the ends of the quartz tube. Teflon ring is used to provide resistance to heat flow from quartz reactor to the Silicone O rings. The stainless steel seals on inlet and outlet of the reactor are also heat isolated from the quartz tube by Teflon disks. The reactants from gas sources are introduced in the reactor through the gas feed lines, which have the outer diameter of 1/4 in. Oxygen, Teos and nitrogen were introduced into the reaction tube through the common gas inlet. The liquid Tetraethyl orthosilicate is contained in the stainless steel bubbler. MFC is used to control the flow of nitrogen gas, which is used as a carrier gas for DMDS in the reactor. We have deposited the SiO₂ films by keeping all processing parameters constant except the deposition temperature. The flow rates of gases to reactor chamber are precisely controlled, because, the control on gas phase and the surface reaction is of inevitable requirement for achieving the better electrical and optical properties of films.

We have supplied Tetraethyl orthosilicate and Oxygen gas simultaneously to the CVD reactor in a controlled manner for the growth of silicon oxide thin films which helps to control over various properties of deposited thin films like composition, stoichiometry, crystallinity, density, rate of deposition of the film, and film morphology. The operation of the thermal-CVD system is controlled through the control panel. The panel consists of the furnace controls for temperature and electric current and voltage. The exit gas line is 1/2 in. in diameter and its end opens in the rotary pump to control pressure into the reactor followed by the gasses scrubber.

The optimized values of the different process parameters that have been used are given in Table 1.

5.2 Process parameters:

Table 1

Process parameters	Physical value
Substrate temperature	700°C to 800°C
Process time	15 min
O ₂ flow rate	3.5 l/min
Nitrogen flow rate with TEOS	2.5 l/min
TEOS bubbler temperature	45°C to 50°C

Chapter 6

Result

6.1 Result:-

Fig. 1 shows the effect of deposition temperature on the refractive index of thermally grown SiO₂ films. It is clearly seen from the figure that the refractive index (RI) decreases in accordance Fig. Schematic diagram of thermal-CVD machine. Table 1 Optimized values of the process parameters used for the deposition of the SiO₂ films Process parameter Physical value Nitrogen flow rate with TEOS & Oxygen (O₂) flow rate 3.5 l/min. Deposition time 15 min Deposition temperatures 700–800 °C with the corresponding increase in deposition temperature. The decrease in the RI may be due to the reaction between oxygen and TEOS.

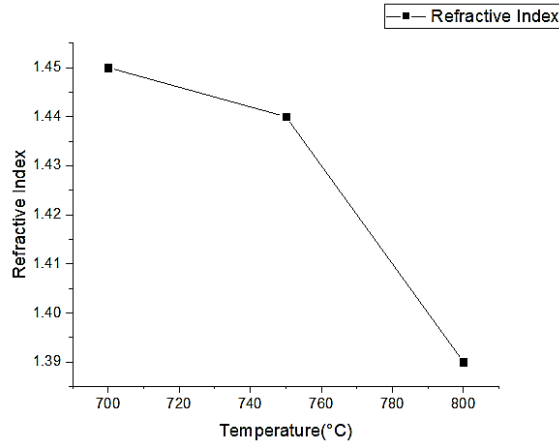


Fig. 1. Effect of deposition temperature on the refractive index of the deposited SiO₂ films.

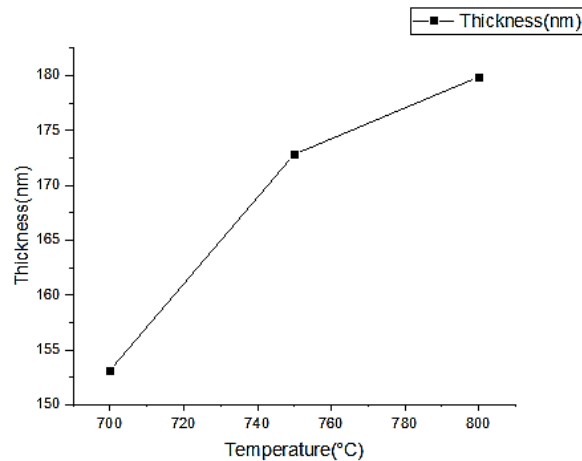


Fig. 2. Effect of deposition temperature on the thickness of the deposited SiO₂ films.

Conclusion

Thermal chemical vapor deposition (TCVD) processes for the deposition of thin-film electronic materials requires an integrated approach involving synthetic chemistry, physical chemistry, and chemical engineering. By refurbishing the TCVD system and implementing necessary updates such as a new Quartz tube, renewed internal wiring, and a Digital Temperature controller (DTC), the system has been successfully optimized for the deposition of SiO₂ thin films. To further enhance the deposition process, understanding and optimization of various process parameters are being carried out. These parameters include substrate temperature, TEOS bubbler temperature, oxygen (O₂) flow rate, TEOS flow rate, and process time. By precisely controlling these parameters, the goal is to achieve uniform and smooth thin film deposition, which is crucial for the desired electronic properties.

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