JAYPEE INSTITUTE OF INFORMATION TECHNOLOGY , NOIDA

B.TECH SEMESTER IV
REPORT PBL
Introduction to Microfabrication Lab (24B25EC212)

GROUP 12
MOS Capacitor Fabrication using Al_2O_3

Submitted by

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INTRODUCTION AND THEORY

The acronym MOS stands for metal–oxide–semiconductor. An MOS capacitor is made of a semiconductor body or substrate, an insulator film, such as SiO2, and a metal electrode called a gate. The oxide film can be as thin as 1.5 nm. One nanometer is equal to 10 Å, or the size of a few oxide molecules. Before 1970, the gate was typically made of metals such as Al (hence the M in MOS). After 1970, heavily doped polycrystalline silicon has been the standard gate material because of its ability to withstand high temperature without reacting with SiO2. But the MOS name stuck. Unless specified otherwise, you may assume that the gate is made of heavily doped, highly conductive, polycrystalline silicon, or poly-Si for short. After 2008, the trend is to reintroduce metal gate and replace SiO2 with more advanced dielectrics for the most advanced transistors

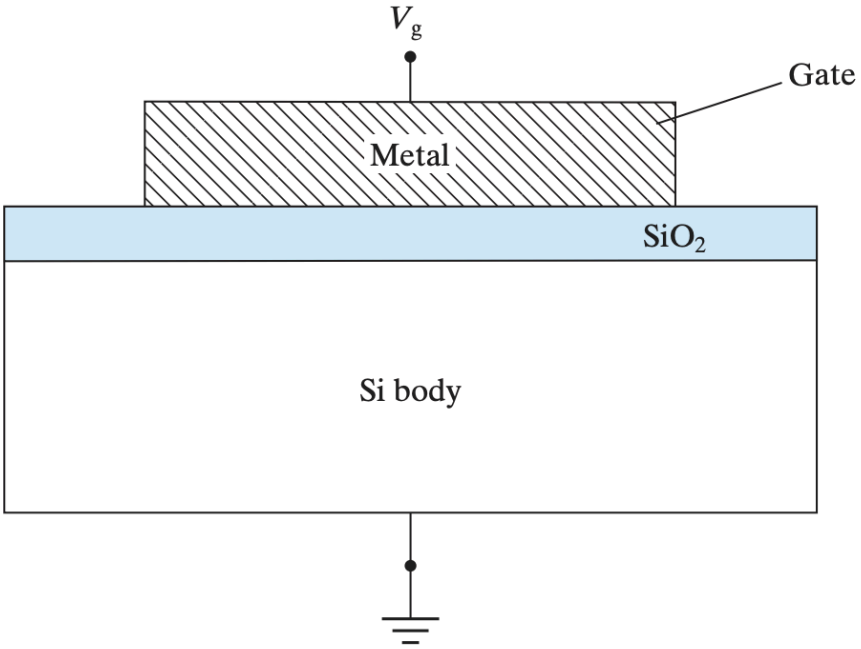

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Figure 1:- The MOS Capacitor

A Metal-Oxide-Semiconductor (MOS) capacitor consists of three layers: a metal gate, an oxide dielectric layer, and a semiconductor substrate. This structure forms the basis for MOSFETs and other semiconductor devices. When a voltage is applied to the gate, the charge distribution in the semiconductor is modulated, leading to three distinct operating regions: accumulation, depletion, and Inversion.

But in this project we will make the MOS capacitor fabrication using Al2O3 deposition . First we will cutting and cleaning the silicon wafer in desired size then we will do Al2O3 deposition using RF Sputtering then we will do the deposition of Aluminium (Al) metals using the thermal deposition then we will do the CV Characterisation using the Keysight Semiconductor Characterization System (SCS).

Cutting and cleaning

Substrate cleaning is a critical step in many material science and nanotechnology applications. Contaminants such as dust, organic residues, and oxides can adversely affect the adhesion, uniformity, and performance of thin films or coatings. Silicon and glass are widely used substrates, and their cleaning requires specific protocols to achieve cleanliness at a microscopic level. Various cleaning methods are employed depending on the type and level of contamination. Here are the common types:

- Radio Corporation of America (RCA) Cleaning
  - Standard Clean (SC-1)
    - SC-1 (Standard Clean 1): Uses a mixture of ammonium hydroxide (NH_4OH), hydrogen peroxide (H_2O_2), and water (H_2O) to remove organic contaminants and particles.
    - H_2O_2 function is oxidize organic contaminants and NH4OH remove the heavy ions: Co, Hg, Ni.
    - SC-1 removes organic residues and set up a condition for desorption of trace metals from the surface .
    - Oxides films keep forming and dissolving .

  - Standard Clean (SC-2)
    - SC-2 (Standard Clean 2): Involves a mixture of hydrochloric acid (HCl), hydrogen peroxide (H_2O_2), and water (H_2O) to remove alkali ions (Na, K, Al, Mn) and hydroxides and complex residual metals.
    - Leaves a protective oxide layer.
    - Order of SC-1 and SC-2 can be reversed.
    - If oxide free surface is required ,HF step is used before , in between ,or after the RCA cleans.

- Piranha Cleaning
    - Piranha cleaning is a popular process for cleaning silicon wafers, but it must be tightly controlled to be effective. Piranha clean, also known as Piranha solution, removes large amounts of organic residues from wafer substrates. It effectively removes photoresist and other hard-to-remove organic materials. A typical mixture of sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) is used.
    - The mixture is a strong oxidising agent , it will removes most organic matter , and will also hydroxylation most surface (add OH groups), making them hydrophilic (water compatible).

➔ Pirana solution should always be prepared by adding hydrogen peroxide to sulphuric acid very slowly surrounding ice cooled reserver ,never vice versa.

➢ HF (Hydrofluoric Acid) Cleaning

➔ HF solution is used to remove native oxide layers from the silicon wafer surface. This is often used before processes that require an oxide-free surface.

- **RF sputtering**

RF or Radio Frequency Sputtering is the technique involved in alternating the electrical potential of the current in the vacuum environment at radio frequencies to avoid a charge building up on certain types of sputtering target materials, which over time can result in arcing into the plasma that spews droplets creating quality control issues on the thin films – and can even lead to the complete cessation of the sputtering of atoms terminating the process. By alternating the electrical potential with RF Sputtering, the surface of the target material can be "cleaned" of a charge buildup with each cycle. On the positive cycle, electrons are attracted to the target material or cathode giving it a negative bias. On the negative portion of the cycle – which is occurring at the radio frequency of 13.56 MHz used internationally for RF power supply equipment – ion bombardment of the target to be sputtered continues .

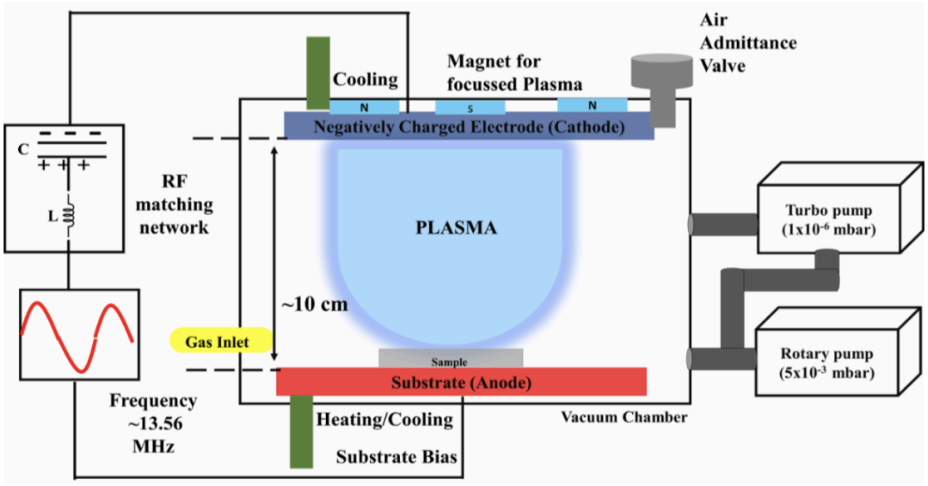


Figure 2:- Schematic for deposition of thin films by RF Sputtering

- **Thermal deposition**

➢ Overview of Thermal Evaporation

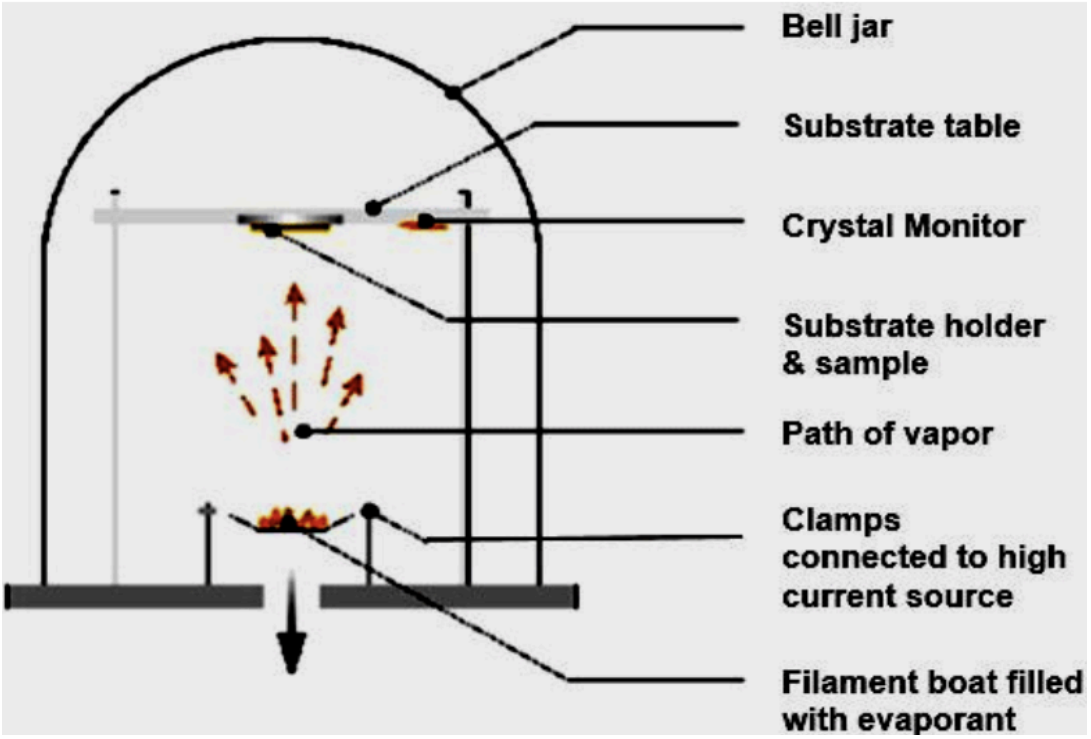


Figure 3:- Schematic of Thermal Evaporator

Thermal evaporation is a Physical Vapor Deposition (PVD) technique that is widely used for depositing thin metal films in semiconductor fabrication. The process involves resistive heating of a metal source in a high-vacuum environment to convert it into vapor, which then condenses onto a cooler substrate, forming a thin film.

The basic principle is based on the relationship between temperature and vapor pressure:

- ➔ When the metal source is heated, its vapor pressure increases.
- ➔ Once the vapor pressure reaches a certain level, metal atoms escape from the source surface into the vacuum.
- ➔ These evaporated atoms travel in a straight-line path in the vacuum and condense onto the substrate surface.

➢ Vacuum System in Thermal Evaporator

A high-vacuum environment is critical in the thermal evaporation process for several reasons:

- ➔ Prevents contamination: Reduces unwanted interactions with atmospheric gases (oxygen, nitrogen).

- ➔ Enhances Mean Free Path (MFP): Ensures that evaporated atoms travel uninterrupted to the substrate.
- ➔ Improves Film Uniformity: High vacuum leads to better adhesion and uniform deposition.
- ➔ Minimizes Oxidation: Reduces oxidation of metals such as aluminium and silver, ensuring high-purity deposition.

The vacuum system consists of:

- ➔ Rotary Pump: Creates an initial vacuum (~ 10−3 Torr).
- ➔ Diffusion Pump: Further reduces pressure to high vacuum levels (10−6 Torr).
- ➔ Vacuum Gauges (Pirani & Penning): Monitor and control the chamber pressure.

➢ Deposition Parameters

- ➔ Vacuum Level: 10−6 to 10−7 Torr
- ➔ Evaporation Temperature: 800°C - 1500°C (depending on metal)
- ➔ Deposition Rate: 1-5 Å/s (Controlled using Quartz Crystal Monitor)
- ➔ Film Thickness: 50 nm - 500 nm (Measured by Thickness Monitor)

● CV characteristics

The Keysight Semiconductor Characterization System (SCS) is an integrated platform designed to perform electrical characterization of semiconductor devices and materials. It combines precision instrumentation, flexible configuration, and powerful software control to measure critical electrical properties such as current-voltage (I–V), capacitance-voltage (C–V), pulse measurements, low-frequency noise, and reliability testing.Common systems include models like the Keysight B1500A, B1505A, and E5270B, each tailored for different voltage, current, and measurement accuracy requirements.

The C-V characterization of the MOS capacitor highlighted the superior performance of Al_2O_3 as a dielectric compared to traditional SiO_2. By applying a bias voltage sweep at 100 KHz (bias voltage of -4 to 4V), we assessed the capacitance behavior across accumulation, depletion, and inversion regions, revealing a well-defined interface with the silicon substrate. Al_2O_3's higher dielectric constant allows for greater capacitance density, enabling thinner dielectric layers without sacrificing charge storage, a significant advantage over SiO_2, which requires thicker layers to prevent leakage. This property supports better device scaling for advanced microelectronics. Additionally, Al_2O_3 exhibits

lower leakage currents due to its robust insulating nature, enhancing device reliability. The smooth C-V transition and minimal noise indicate a cleaner interface with fewer traps compared to SiO_2, which often suffers from higher defect densities. Furthermore, Al_2O_3's thermal stability during fabrication ensures consistent performance, unlike SiO_2, which can degrade under high-temperature processing. These characteristics position Al_2O_3 as a preferable dielectric for next-generation MOS devices, offering improved efficiency and performance in modern semiconductor applications

PROCESS / METHODOLOGY

- **Cutting and Cleaning**
  - ➢ Wafer cutting
    - ➔ Cut the silicon wafer as per desired size.
    - ➔ Use diamond cutter for silicon wafer.

  - ➢ Initial Rinse
    - ➔ Rinse the silicon wafer thoroughly with DI water to remove loose particulates.
    - ➔ Use a clean pair of Teflon-coated tongs to handle the substrates to avoid any contamination.
    - ➔ Use a teflon support clean holder to hold the substrate for Cleaning the silicon substrate (because teflon holder does not react with acid).

  - ➢ Piranha Solution Cleaning (for Silicon Substrates)
    - ➔ Prepare the piranha solution by carefully mixing 3:1 concentrated sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) in a beaker. Always add peroxide to acid, not the other way around .
    - ➔ Submerge the silicon substrate in the piranha solution for 10 minutes to remove organic and inorganic contaminants.
    - ➔ Rinse the substrate thoroughly with DI water2 or 3 times.

  - ➢ RCA-1 (Radio Corporation of America 1) or SC-1 (Standard Clean 1)
    - ➔ Prepare the RCA-1 or SC-1 by carefully mixing 7:1:2 concentration of distilled water, ammonium hydroxide (NH4OH) and hydrogen peroxide (H_2O_2) in a beaker . Always add peroxide to acid ,not the other way around.
    - ➔ Submerge the silicon substrate in the RCA-1 solution for 10-15 min at the temperature of 85 degree celsius
    - ➔ Rinse the substrate thoroughly with DI water2 or 3 times.

  - ➢ RCA-2(Radio Corporation of America 2) or SC 2 (Standard Clean 2)
    - ➔ Prepare the RCA-2 or SC-2 by carefully mixing 7:1:2 concentration of distilled water, hydrochloric acid (HCl) and hydrogen peroxide (H_2O_2) in a beaker . Always add peroxide to acid ,not the other way around.
    - ➔ Submerge the silicon substrate in the rca2 solution for 10-15 min at the temperature of 85 degree celsius
    - ➔ Rinse the substrate thoroughly with DI water2 or 3 times.

➢ HF (Hydrofluoric Acid) Cleaning (for silicon substrate)

- ➔ Prepare the mixture of HF and Di water 1:10 in teflon weaker.(As HF solution reacts with glass beaker but teflon is highly resistant for HF solution)
- ➔ Carefully immerse the silicon wafers into the HF solution using tongs or wafer tweezers.
- ➔ Dip the wafers for a duration typically 5 minutes to remove the native oxides.
- ➔ Rinse the substrate thoroughly with DI water2 or 3 times.
- ➔ Check the wafer if the water holds in the wafer or not ,if the distilled wafer not holds the water that means the wafer cleaning is done properly , if wafer holds the DI water then we have to clean the wafer once again using HF solution.
- ➔ Dry the substrate using nitrogen gas or oven at 90’C for 10 min only.

➢ Inspection

- ➔ Inspect the substrate surface under a microscope to confirm cleanliness. There should be no visible residues or particles.
- ➔ If residues persist, repeat the cleaning process as necessary.

➢ Storage

- ➔ Store cleaned substrates in a clean, dry environment, such as a sealed container or desiccator, to prevent contamination.

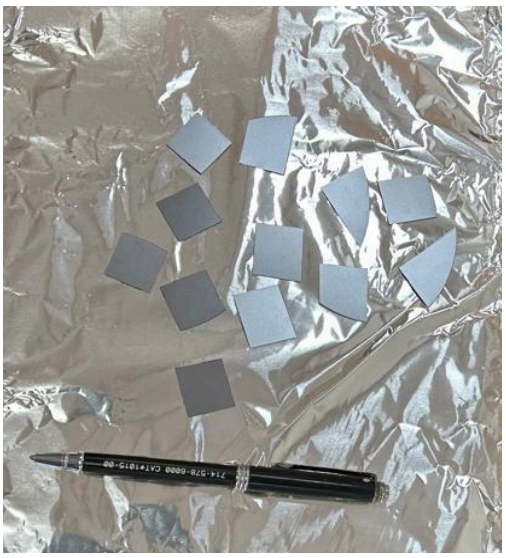


Figure 4 :- Cutting Substrate

Figure 5 :- Cleaned Substrate

- **Deposition of Al2O3 using RF Sputtering**
  - ➔ Turn on the machine, Air compressor, and chiller.
  - ➔ Vent the chamber .
  - ➔ Remove the substrate holder and target holder .
  - ➔ Load the target in the target holder.
  - ➔ Mount the substrate onto the substrate holder using Krypton tape.
  - ➔ Turn the Reset Button OFF (beneath the Turn-On button).
  - ➔ Press the Vent Button OFF to start the rotary pump.
  - ➔ Turn the Reset Button OFF (beneath the Turn-On button).
  - ➔ Turbo pump starts accelerating. Wait until the Turbo Pump Ready signal shows.
  - ➔ Once the turbo pump is ready, turn ON the HV (High Vacuum) Pump.
  - ➔ Wait until the desired vacuum (e.g., 10^{-6} Torr) is reached.(we have started vacuum cracking when the pressure reaches to $5.2x10^{-6}$).
  - ➔ Press the Process button to start vacuum cracking (system prepares for deposition).
  - ➔ Go to the Process Screen and turn ON the gas valve.
  - ➔ Slowly increase the gas of argon and oxygen .(usually in sccm(standard cubic per minute)).
  - ➔ Slowly increase the RF set power . Till the reflected power does not became zero. (RF power depend on material).
  - ➔ After the deposition time is complete, turn OFF the RF power first.
  - ➔ Close the gas valve.
  - ➔ Press Process Off or Deposition Stop.
  - ➔ Turn OFF the HV Pump .
  - ➔ Vent the chamber (slowly) to bring it back to atmospheric pressure.
  - ➔ Remove the substrate carefully.

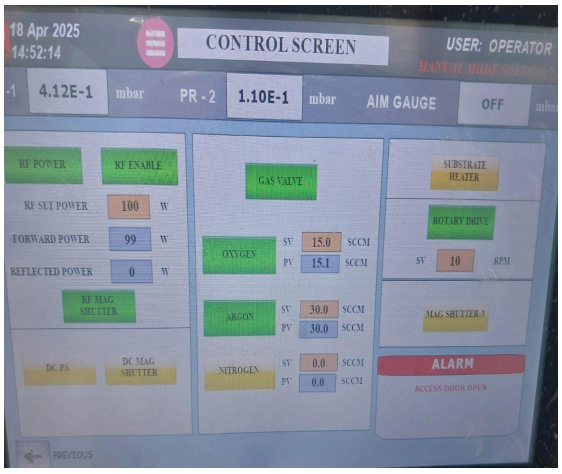


Figure 6 :- Control Screen of RF Sputtering Figure 7 :- Display Screen Of RF Sputtering

- **Deposition of Al using Thermal Evaporation**
  - ➢ System Preparation
    - ➔ Ensure Cleanliness: Wipe the vacuum chamber, substrate holder, and metal source to remove contaminants.
    - ➔ Remove the substrate holder from the machine ,and
    - ➔ Load Metal Source: Place the metal pellets (Al, Au, Ag) in the tungsten/molybdenum boat.
    - ➔ Mount the Substrate: Secure the silicon/glass wafer on the substrate holder.
    - ➔ Close the Chamber: Ensure proper sealing to maintain vacuum integrity.

  - ➢ Vacuum System Operation
    - ➔ Start the Rotary Pump: Achieve initial vacuum (~ 10−3 Torr).
    - ➔ Activate Diffusion Pump: Reduce pressure further (10^{-6} Torr by keeping the rotary pump in backing.(we have done the vacuum pressure till $2.7x10^{-6}$).
    - ➔ Monitor Vacuum Gauges: Ensure pressure stability before proceeding.
    - * Pre heat the oil in the diffusion pump (for ~1 hours) so that vacuum formation is not hindered. The system will give indication when the pump is ready to use.

  - ➢ Heating and Deposition
    - ➔ Increase Current to Filament: Heat the metal until evaporation starts (~ 800°C for Al, ~ 1100°C for Au).
    - ➔ Monitor Deposition Rate: Use the Quartz Crystal Monitor to control the rate (1-5 Å/s).
    - ➔ Control Film Thickness: Stop heating when the required thickness is achieved (~ 100nm).

  - ➢ Cooling and Chamber Venting
    - ➔ Allow System to Cool: Prevent oxidation or damage to the film.
    - ➔ Vent the Chamber Slowly: Introduce air to the contamination.
    - ➔ Carefully Remove the Substrate: Observe the deposited thin film.

Figure 8:- Vacuum Observation in Thermal Evaporation

Figure 9 :- Vacuum Chamber of Thermal Evaporator

- **C-V Characterisation using Keysight Semiconductor Characterization System (SCS).**

➢ Setup and Connections

➔ Mount the MOS capacitor sample onto the probe station.

➔ Connect the Keysight Semiconductor Characterization System as follows:

- ☐ SMU1 (Source Measurement Unit 1) → Gate terminal (Metal layer)
- ☐ SMU2 (Ground) → Substrate (Semiconductor layer)

➔ Ensure proper shielding and grounding to minimize noise and leakage currents.

➔ Configure the Keysight Easy EXPERT software for measurement.

➢ C-V Measurement Using Keysight SCS

➔ Open Keysight EasyEXPERT Software on the system.

➔ Select Capacitance-Voltage (C-V) measurement mode.

➔ Configure the test parameters:

- ☐ Voltage sweep range: -4V to 43V (for p-type substrate).(It depend on you in which ranges you are selected).
- ☐ Measurement frequency: 100 kHz (for high-frequency C-V).(It depends on you at what frequency you need to plot cv graph).
- ☐ Step size: 50 mV. (Automatically measured on the basis of voltage range and frequency).

➔ Run the measurement and observe the C-V curve.

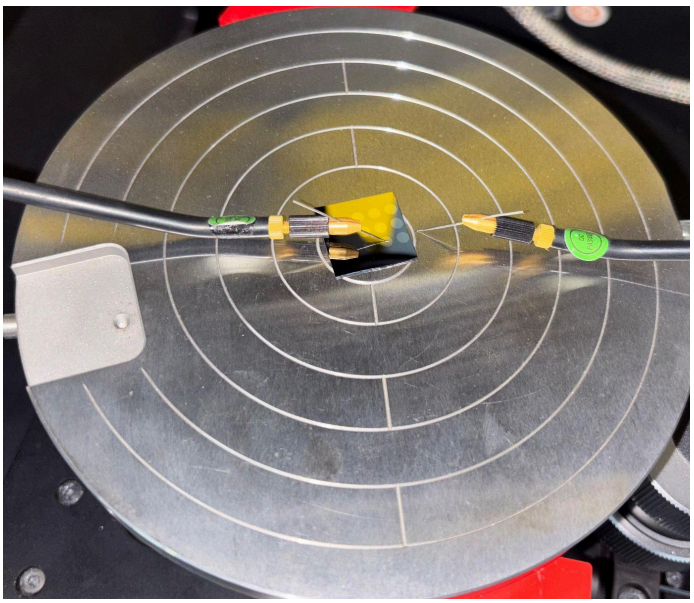


Figure 10 :- Substrate on Keysight SCS probe station

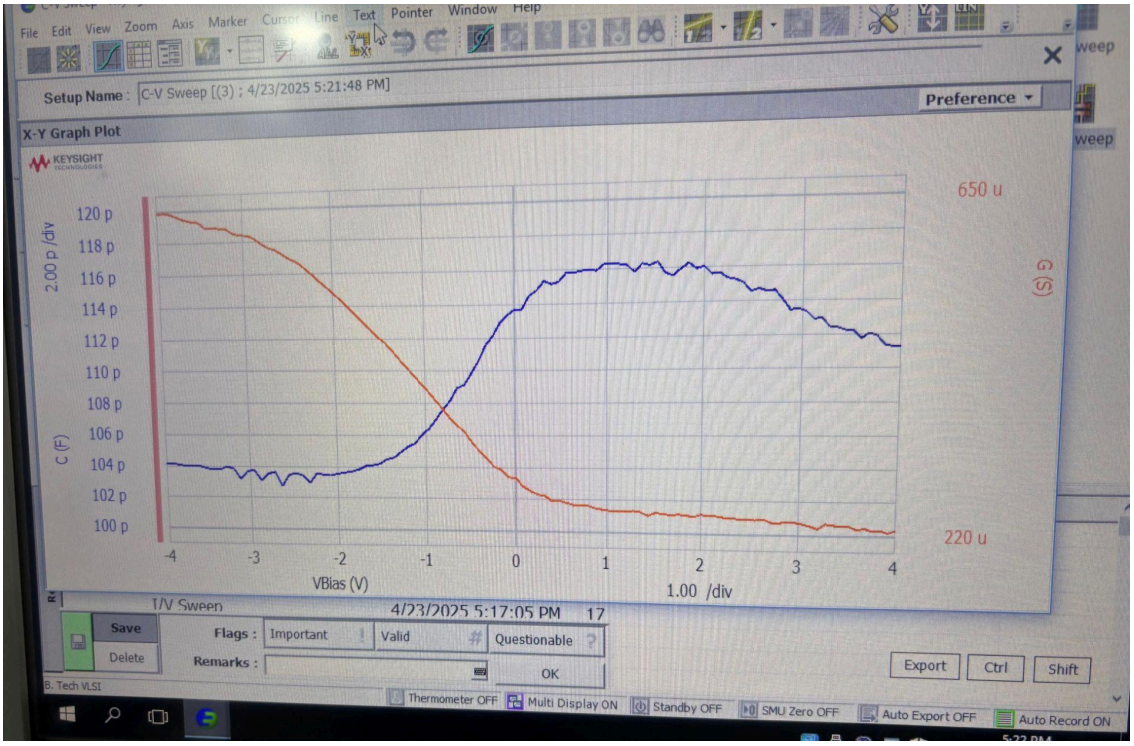
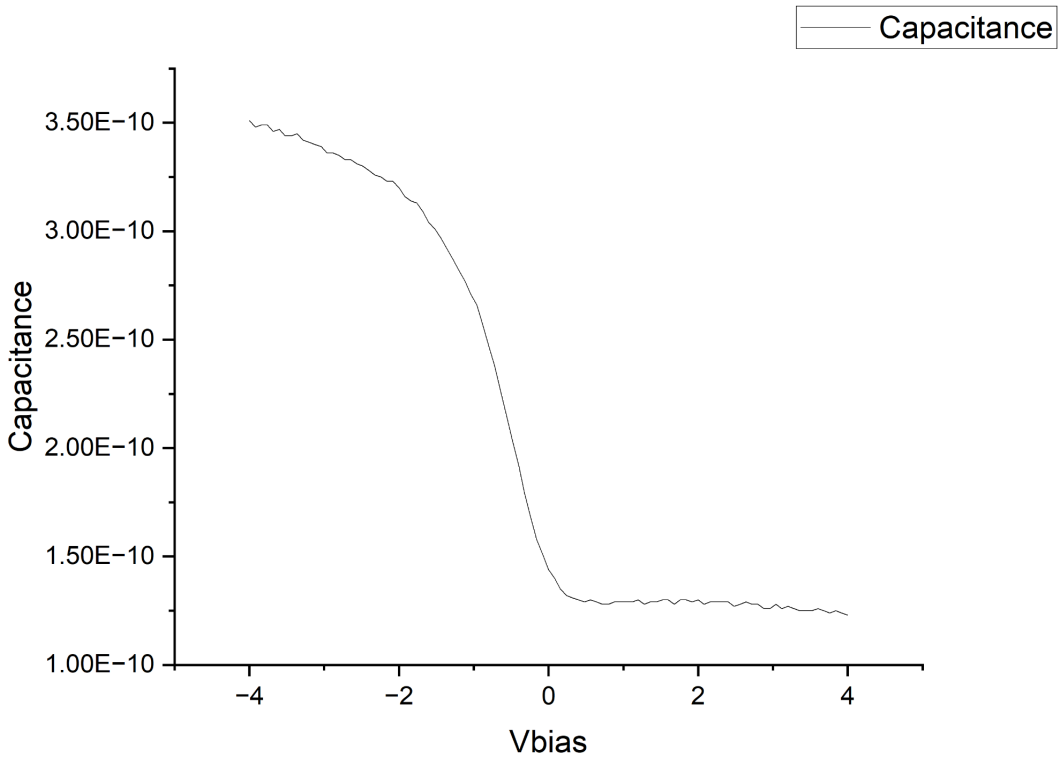


Figure 11:- Output of C-V Curve on Keysight SCS Screen



Graph 1- Demonstrating the C-V curve of fabricated MOS Capacitor using Origin Software

RESULT AND DISCUSSION

The MOS capacitor fabricated with Al_2O_3 as the dielectric layer exhibited promising electrical performance, as evidenced by the capacitance-voltage (C-V) characteristics shown in the graph. The C-V curve demonstrates a clear transition from accumulation to depletion, with capacitance dropping from 3.5×10^{-10} F to 1.0×10^{-10} F as the bias voltage (V_bias) sweeps from -4 V to 4 V at frequency of 100K hz. This behavior indicates a well-formed Si-Al_2O_3 interface.

Major parameters extracted from graph:

Threshold voltage- 0.88V at 1.29×10^{-10} F

Flat band voltage- 1.29V at 3.01×10^{-10} F

Cmin - 1.23×10^{-10} F at 4V (inversion region)

Cox- 3.51×10^{-10} F at -4V (accumulation region)

The observed C-V characteristics reflect the quality of the Al_2O_3 dielectric and the precision of the fabrication process. The sharp capacitance drop with increasing V_bias suggests a low density of interface traps, likely due to the controlled RF sputtering parameters, such as power and deposition time, which ensured a dense and uniform Al_2O_3 layer. Thermal evaporation of the aluminum contacts provided robust electrical connectivity, minimizing contact resistance. However, the slight noise in the C-V curve at higher voltages indicates possible minor defects in the dielectric, which could be addressed through post-deposition annealing. Overall, these results validate the suitability of RF sputtering and thermal evaporation for fabricating high-performance MOS capacitors with Al_2O_3 for advanced microelectronic applications.

CONCLUSION

The fabrication of the MOS capacitor with Al_2O_3 involved several critical stages, beginning with thorough substrate cleaning and preparation to ensure a pristine surface. This was followed by RF sputtering to deposit the Al_2O_3 dielectric layer, with precise control over parameters like power, pressure, and deposition time to achieve a uniform film. Subsequently, thermal evaporation was employed to deposit aluminum contacts, ensuring low-resistance electrical connections. The process concluded withC-V characterization, which revealed a capacitance range from 3.5×10^{-10}F to 1.0×10^{-10} F across a -4 V to 4 V bias sweep, demonstrating a robust Si-Al_2O_3interface and validating the effectiveness of the fabrication techniques.

The output results highlight the capacitor's potential for advanced microelectronic applications, with a low interface trap density and capacitance behavior aligning with theoretical expectations for Al_2O_3 as a high-k dielectric. The slight noise observed in the C-V curve at higher voltages suggests minor defects, likely due to imperfections in the dielectric layer, which could be mitigated through post-deposition annealing or refined sputtering conditions. Overall, this study successfully demonstrates a viable fabrication process, with the capacitor showing promise for low-power, high-performance devices. Further optimization could enhance its reliability and scalability in future semiconductor technologies.