CHAPTER 3

Fabrication and Characterization of Gridded Gate Pt/SiO_2/Si MOS Sensor

3.1 Introduction

This chapter deals with experimental techniques used for the fabrication and characterization of MOS based hydrogen gas sensors. Initially, a brief description of the facilities available at our laboratories has been presented followed by a detailed description of fabrication process adopted for gridded Platinum gate MOS capacitor sensors.

3.2 Facilities Available in the Laboratory

3.2.1 Vacuum Coating Unit

The Vacuum coating unit model no.: 12A4D (Hindhivac Co. Ltd., India, make) is illustrated in Plate-3.1. The Vacuum chamber is made of electrochemically- polished stainless steel. The Vacuum chamber consists three circular glass windows which enables visual inspection of the ongoing coating process. The chamber is placed on the base plate, makes a vacuum tight seal with the base plate by means of an 'L' type neoprene gasket. The chamber is evacuated by a diffusion pump backed by a 250 litre/min., double stage direct driven rotary vacuum pump having an overload protection. The pressure measurements are made by the pirani gauge head in the range 0.5 torr to 10^{-3} torr and by the penning gauge in the range 10^{-3} torr to 10^{-6} torr. The pirani gauge is used to measure roughing and backing pressure and the penning gauge is used to measure lower pressure in the chamber. Five different vacuum valves are fitted with the system as described below:

1. A hand operated high vacuum valve is fixed to the 13 inch diameter base plate. This valve isolates the chamber from the pumping system so that the chamber can be brought to atmospheric pressure without switching off the pumping system.

2. Two quarter swing butterfly type valves are provided, one in the backing line and other in the roughing line. The backing valve isolates the diffusion pump from the rest of the system when roughing is ON. When high vacuum pump is 'ON', roughing valve is closed and the backing valve opened to allow the rotary vacuum pump to back the diffusion pump.

3. A 3/4 inch port diaphragm isolation valve, straight through type, is provided in the rotary vacuum pump line between the pump and the pirani gauge head for throttling the flow of gases to the rotary pump for leak detection purpose.

Plate3.1: Vacuum Coating Unit (model no.: 12A4D, HindhiVac Co. Ltd.)

4. A 1 inch port magnetic isolation cum air admittance valve is provided in the vacuum pipeline above the rotary pump. When the unit is switched off or power supply fails, the valve isolates the entire system and admits air to the rotary pump. Contamination of pipelines with rotary oil is thus prevented.

5. A 1/4 inch air admittance valve is fixed to the chamber pipeline to release the chamber vacuum after a coating process. The chamber is provided with a work holder ring of 8 inch diameter. The ring is supported by three pillars fixed in the base plate. A standard filament holder is fixed to the L.T. drive electrode and an earth electrode. The filament is normally positioned vertically below the center of the work holder to give uniform distribution of the evaporation. Three sets of off-center filament holders are supplied to fix them to three sets of L.T. electrodes for multilayer coating. An 80 amps rotary switch is provided to select any one of the three L.T. electrodes.

3.2.2 Diffusion Furnace

The "mini-Brute" bench model furnace (model no.: MB71,Thermco Inc., USA) is illustrated in Plate 3.2. It has three independent furnaces;

1) The bottom furnace for oxidation 2) the middle furnace for p-diffusion

3) The top furnace for n-diffusion.

It also consists a Ana-lock (model 431) general purpose controller with a temperature range of 200 ^{o}C to 1200 ^{o}C. It is fitted with process tubes of 3.25 inch outer diameter which has a uniformly heated flat zone of 14 inches with ±0.5 ^{o}C accuracy. The heating element itself is a three zone continuously wound helical coil, manufactured of #2 gauge nickel-chrome-aluminium wire mounted in low K factor insulating material. The furnace is equipped with copper tube for water cooling facility. Water cooling to the heating chamber removes approximately 60% of the BTU (British Thermal Unit) required to operate the furnace which would otherwise dissipate into the room. Besides water cooling, air cooling is also done through silent running, high volume circulating fans, which draw the room air to keep all the running components trouble free even at the maximum operating temperature rating.

Plate 3.2: Diffusion Furnace

3.2.3 Thermo Chuck

The Thermo chuck (model TP36, Temptronics Corporation make) has been illustrated in Plate 3.3**,** consisting a controller and a temperature controlled thermo-chuck. Temperature control is obtained by an AC zero cross with the help of thermister sensing of temperature, coupled to the thermo-chuck surface. The low and high temperature can be adjusted with the help of an indicator. A timer is also provided on the front panel, which can hold the temperature for a particular time and where the time is elapsed set by the timer, cooling begins automatically in auto mode of operation. This model has an accuracy of ±6 ^{o}C, stability of ±1% of the chuck temperature (^{o}C) and time range of 0.2 sec-60 hrs value.

Plate 3.3: Thermo Chuck

3.2.4 Photolithography Set-Up

The facilities used for the photolithography is shown in Plate 3.4**.** It includes photo-resist spinner provided with a vacuum chuck for substrate holding, speed controller and timer. Spin Coater (model no. SPM 150LC, TSE systems make) is shown in Plate 3.4 (a). To expose the thin layer of photoresist on the wafer, the ultra-violet light was used. The Set-up for UV exposure is shown in Fig. 3.4 (b).

(a)

(b)

Plate 3.4: Photolithography Set-up **(a)** Spin Coater **(b)** Set-up for UV exposure

3.2.5 RF Plasma Enhanced Chemical Vapour Deposition System (RFPECVD)

The schematic diagram of PECVD system (courtesy: National Physical Laboratory (NPL), New Delhi, India) has been illustrated in Fig. 3.1, is a parallel plate capacitively coupled system, consisting of bottom circular electrode 4 inch in diameter and the electrode distance varies from 10-12 mm. The throttle valve is provided to control the electrode pressure of the chamber. The top electrode (anode) is grounded and substrate is kept at bottom electrode (cathode) which is fed by RF source that works at 13.56 MHz. After that oxygen is introduced in the process chamber at a flow rate of 99 sccm/minutes keeping the base pressure and working pressure at $2.5x10^{-3}$ and $2.5x10^{-2}$ mbar, respectively. The upper surface of samples was bombarded by RF oxygen plasma. The bottom electrode was driven by varying RF power (40W & 50W) for different time duration (2 min., 4 min., 8 min. & 12 min.) for different samples.

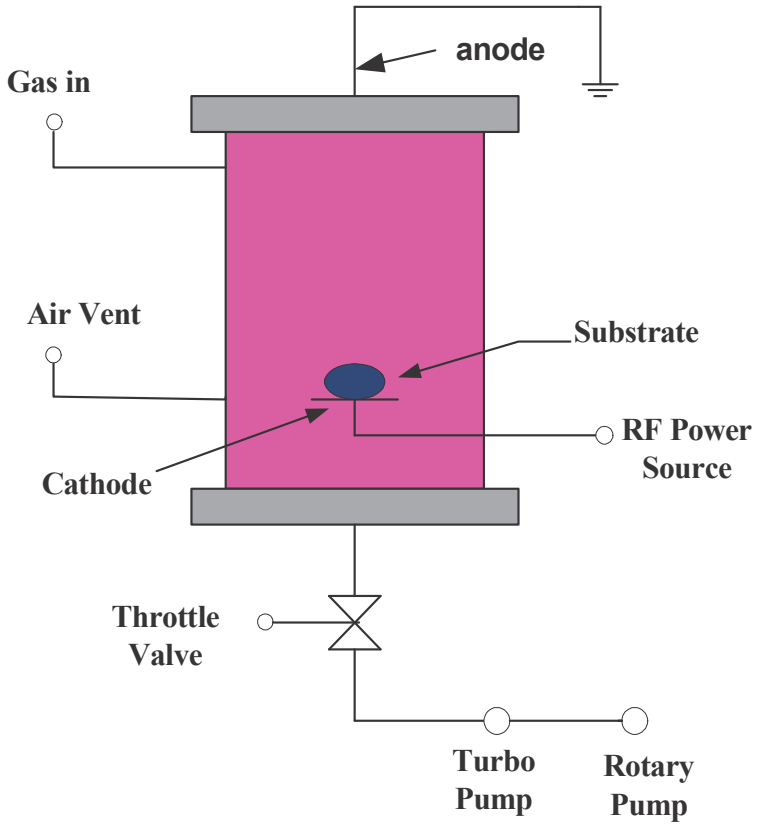

Fig. 3.1 Schematic of PECVD system

3.2.6 SEM and AFM Microscopy

The Scanning electron microscopy (SEM) is an important tool to investigate the surface and morphology of nanostructures. It is used to estimate the diameter, length, thickness, density, shape and orientation of the nanostructures. The microstructural study of the Pt film was carried out by using scanning electron microscope (Quanta 200F; courtesy: Deptt. of Metallurgy Engineering, IIT(BHU), Varanasi, India). The unique advantage of a SEM over other imaging techniques is its huge depth of fields with possible magnification from 10 to 500,000 times.

Atomic force microscopy (AFM) is a technique for analyzing the morphology of a surface. A mechanical probe oscillates close to the sample surface and a feedback mechanism maintains the movement of the tip, the movement of tip is stored. In this way it is possible to obtain high resolution topographical information. The data output is either a three dimensional image of the surface or a line profile with height measurements. AFM typically does some damage to the sample but it is convenient to use since it can be operated in air. In this work, AFM (model no.-NSE, Nanoscope E Digital Instrument Inc., U.S.A; courtesy: UGC-DAE consortium for scientific research, Indore, India) was used in contact mode to investigate the microstructure of the metallic gate film (Pt) and also the plasma treated SiO_2 surface.

3.2.7 Measurement Equipments

3.2.7.1 Manual Ellipsometer

Ellipsometer, model L117, (Gaertner Scientific Corp., Chicago, USA) is used for measuring the surface film thickness and refractive index of films ranging from a few Angstrom to thick films. A production ellipsometer is shown in the Plate 3.5. The light source is a He-Ne 6328 Å laser of 2 mW power which projects 1mm diameter spot on the film surface. The angles of incidence could be chosen to be 30^0, 50^0 and 70^0. The extinction meter or detector has a built-in solid state amplifier. The polarizing optics is Glan-Thompson prism of high quality, extremely smooth, shake free rotary mounts with clear precise graduations. The specimen is placed horizontally and the light spot could be magnified 20 times with a microscope. The light from He-Ne laser is first linearly polarized by passing through the compensator. When the light reflects

from the specimen, the polarization of light changes in accordance to the specimen film thickness, optical characteristics of the film and substrate, this light then passes through the analyzer and is sensed by the photodetector**.** A filter in-front of the photo detector eliminates unwanted background light so that precise measurements can be made at normal room conditions. The amount of the laser light reaching to the photodetector is indicated by extinction-meter. At a certain polarizer setting, the analyzer is rotated to a position where almost no light reaches the photodetector and this results the measurement condition.

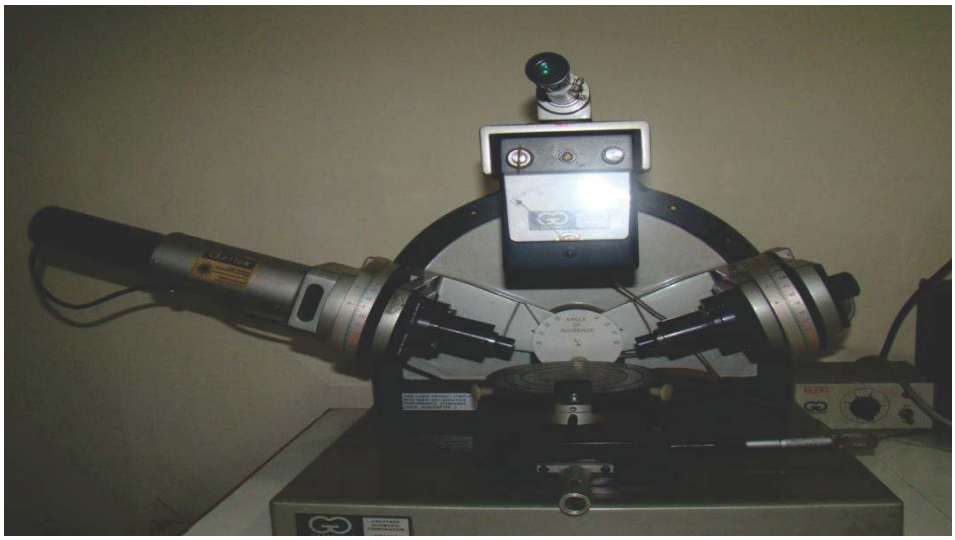

Plate 3.5: Manual Ellipsometer

3.2.7.2 Precision LCR Meter

LCR meter model (HP 4284A) is shown in Plate 3.6**.** This instrument is used for evaluating LCR components, materials and semiconductor devices over a wide range of frequencies (20 Hz to 1 MHz) and test signal levels (5 mV to 2 V r.m.s, 50 µA to 20 mA r.m.s).The instrument sweep function permits the entry of up to ten frequencies, test signal levels, or bias level points to be automatically measured. It has in-built HP-IB having interfaceble capability.

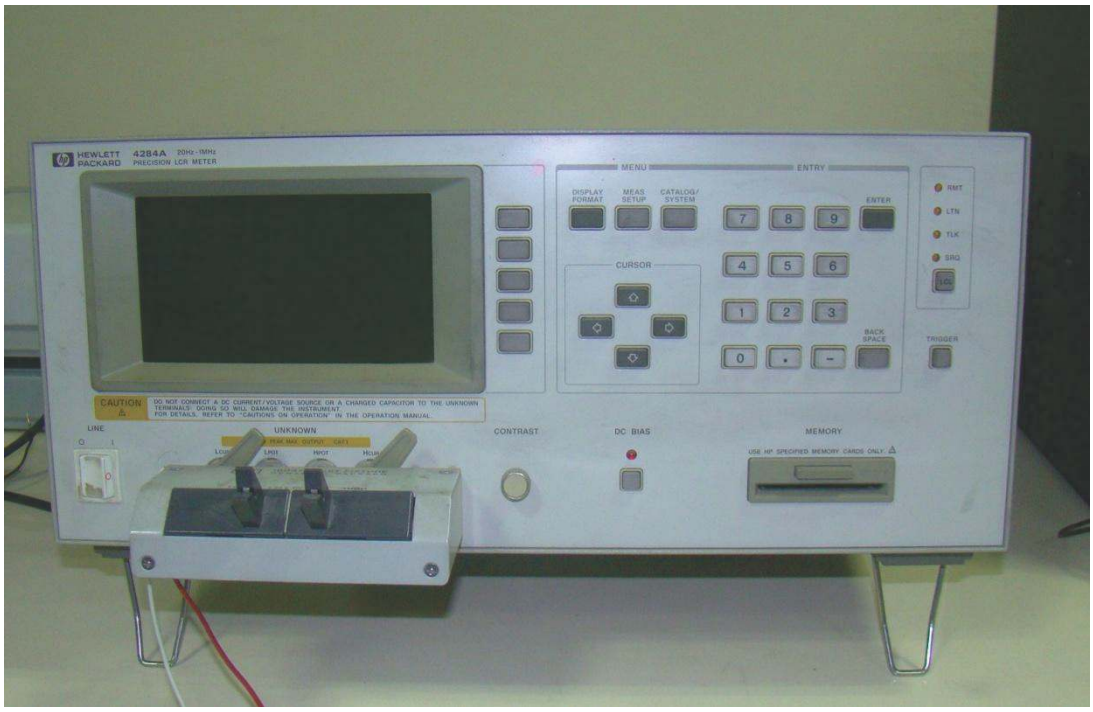

Plate 3.6: Precision LCR meter (model : HP 4284A)

3.3 Developed Facilities

3.3.1 PC Controlled Measurement Set-up

To study the performance of fabricated MOS sensors a test chamber has been designed, developed and successfully installed along with precision LCR meter (model no: HP-4194A, Hewlett packerd make) in our laboratory. Developed test chamber is essentially a closed chamber having vacuum facility. The chamber has the facility of injection of desired volume of gasses. There is also a provision for injection of organic vapors from the top of the chamber through a air tight rubber seal. A small fan (operated with 5 Volt dc source) is also mounted on the top of chamber for evaporating proper mixing of the test gas. The PC controlled measurement set-up is shown in Plate 3.7. The LCR meter (HP-4284A) described in the previous section has inbuilt HPIB interfaceble capacity. This feature of interfacebility was used to control the instruments remotely and gather all the necessary information required for further processing. The LCR meter is interfaced to PC via a GPIB-card of IEEE-488 standard. Interactive Characterization Software (ICS) was installed to obtain the accurate information from the instruments and stored in the computer. Print out of stored data was taken out through printer. To control and measurement of gas flow inside the test chamber rotameter (MARCH instruments, GCM-100, USA mak) was calibrated and used for test gases. To test the sensor for various concentrations, gas injection port at the top of the test chamber was used. Block diagram of the measurement set-up is shown in Fig. 3.2.

Plate 3.7 PC controlled measurement Set-Up

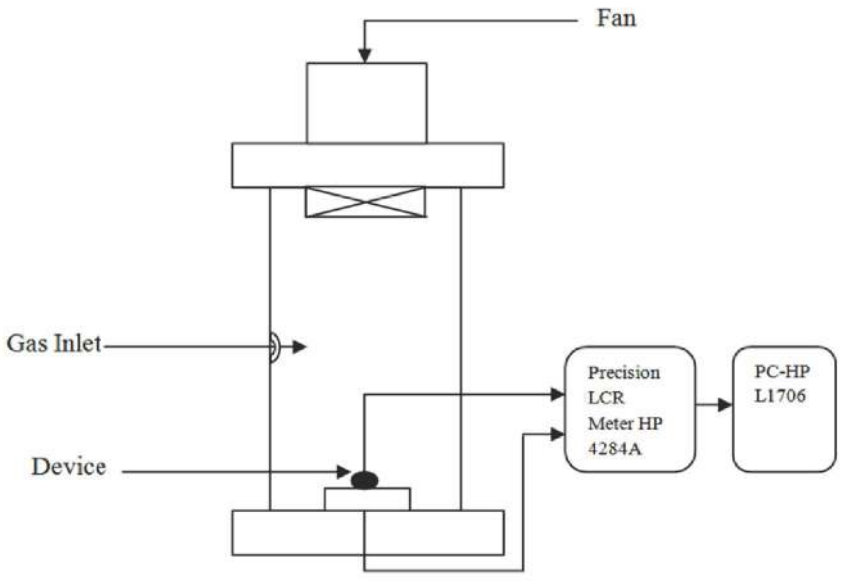

Fig. 3.2 Schematic diagram of developed C-V measurement set-up

3.4 Fabrication of Gridded Pt Gate MOS Gas Sensor

The section describes the specification of Silicon wafer and subsequent processing steps for the fabrication of gridded Pt/SiO_2/Si MOS sensor.

3.4.1 Substrate Specifications

Substrate: Silicon (single crystal)

Make: Wacker Chem., West Germany.

Surface: Polished (one side)

Type: p-type <100>

Resistivity: 3-5 Ω-cm

Thickness: 300-350 μm

Diameter: 75±2 mm

A flow chart representation of the experimental process adopted for the fabrication of the sensor is shown below:

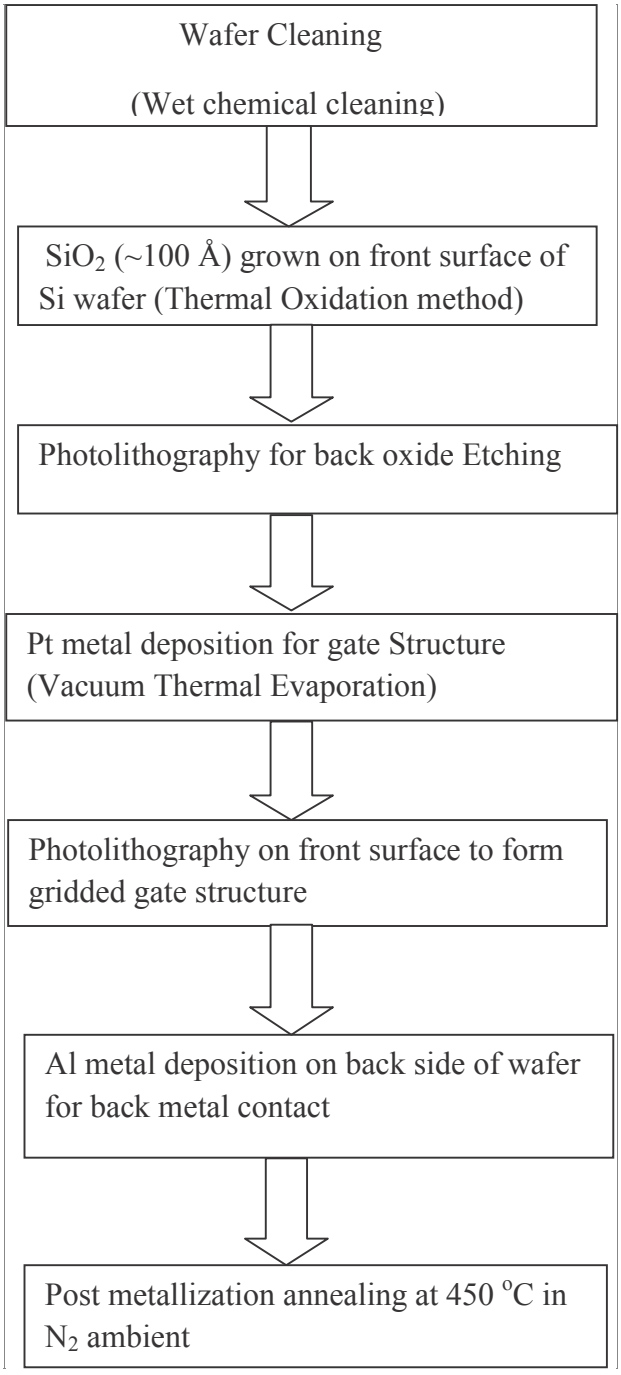

Fig. 3.3 Flow chart of fabrication steps of gridded gate MOS sensors

3.4.2 Substrate Cleaning

The very first step of device fabrication is the cleaning step which is important as it affect the electrical characteristics of device. The substrate cleaning steps which are generally involved are as follows:

i. An ultrasonic agitation is given to the wafer in trichloroethylene for about 5 minutes, and dried subsequently. Such a cleaning step is required to remove the contaminants produced during the process of cutting, lapping, polishing and packaging of the wafers.

ii. The wafer is then rinsed with acetone for 1-2 minutes to remove oil and organic residue that appears on glossy surface.

iii. Further, the wafer is rinsed 4-5 times in DI water of 22 MΩ resistivity.

iv. After drying, the wafer is then rinsed in a solution consisting of H_2SO_4 (40%) and H_2O_2 (60%) for 5 minutes and then thoroughly cleaned in the running deionised water.

v. Wafer is then washed with running DI water 2-3 times.

vi. The wafer is then dipped in HF+DI (1:6) solution for 1 minute followed by 10-15 times with water rinse.

vii. Then, wafer was washed 10-15 times with DI water.

viii. The wafer is then kept in an oven at 100 ^{0}C for 10 minutes enabling the wafer to dry completely.

3.4.3 Growth of Silicon Dioxide (SiO_2) Layer

The furnace illustrated in Plate 3.2 was used to perform the dry oxidation thermally on the substrate. The thermal oxidation using dry oxygen involves the control of the flow of oxygen into the Quartz tube of the furnace to ensure that an excess of oxygen is available for the silicon to oxidize. An oxygen /nitrogen mixture (3:1) ratio is used for growing the silicon dioxide layer on the silicon substrate. The dry oxidation takes place when the oxygen reacts with the silicon substrate as given by the following chemical equation:

$Si + O_2 \rightarrow SiO_2$

The thickness of the oxide layer grown is determined by utilizing the ellipsometry technique which is shown in Plate 3.5. To get the required temperature for the oxidation, the furnace was switched ON and the water was allowed to circulate around the furnace for cooling. The dial of the temperature controller was adjusted to get the required temperature at the central zone of the furnace and the nitrogen gas was introduced into the tube to create an inert atmosphere. The flow rate of the nitrogen gas was controlled at 0.6 lit/min. by the flowmeter attached to the furnace. After attaining the required stable temperature in the central zone of the furnace, the wafers were kept inside the uniformly heated flat zone of the furnace and oxygen was passed through the furnace by controlling the flow rate of 1.8 ltr/min. by other flowmeter which is attached to the furnace for dry oxidation. These two gases i.e. N_2 and O_2 were introduced inside the process tube for calculated time to achieve the desired oxide thickness. Just after completing the oxidation, the oxygen gas flow was stopped completely and the heating element of the furnace was also switched OFF. The flow of the nitrogen is still continued until the furnace temperature comes down to the room temperature.

3.4.4 Photolithography for Back Oxide Etching

The front sides of the wafer were spin coated with negative photoresist using the spinner and then the wafer were prebaked for 10 minutes at 90 ^oC. After prebaking, the surfaces (coated with photoresist) were exposed to UV light for 2 minutes which hardens the photoresist. Then the photoresist is developed in the developer solution and no photoresist is dissolved due to of the hardening of the entire front side of the wafers. Now, the wafers is post baked in the oven for about 20 minutes at 150 ^oC. The complete lithographic process is carried out in the yellow room. After the photolithography process is over the wafers were rinsed in the solution consisting of 6 parts by volume of deionized water and 1 part by volume of HF for about 1 minute to etch out the back oxide. Then, these wafers are cleaned thoroughly in deionised water and dried in hot nitrogen ambient at 200 ^oC. Subsequently, the photoresist from the front side is removed by chemical etching (using the solution consisting of 9 parts by volume of H_2SO_4 and 1 part by volume of HNO_3).

3.4.5 Platinum Metal Deposition for Gate Structure

Platinum (Pt) metallization is done on the front side of the wafer for making ohmic contact. The vacuum coating unit illustrated in Plate 3.1 was used for metallization. Initially, Si substrates with thermally grown SiO_2 were placed in the chamber of coating unit in such a way that the front side faces the filament and rough surface upward. Around 0.5 gm platinum (Pt) metal was kept in the helical tungsten filament and chamber was closed keeping all the valves closed, the rotary pump was started and the pirani gauge head was switched ON. When the pressure inside the chamber reduced to 10^{-2} torr, the backing valve was opened and vacuum was created inside the diffusion pump to about 10^{-2} torr. Now the backing valve was closed and the roughing valve was opened. At the same time, cooling water was admitted to the diffusion pump and the pump was switched ON. When the chamber vacuum reaches of the order 10^{-3} torr by rotary pump, the roughing was closed and the backing valve was opened. Subsequently, the baffle valve was opened slowly and the penning gauge was switched ON. When the vacuum of the order of $0.5x10^{-5}$-10^{-6} torr is achieved in the chamber, the power source is turned on and the current was slowly increased to heat the filament. The Platinum film of average thickness 350Å has been deposited on front face of Si wafer. The thickness of the deposited film was measured by film thickness monitor facilitated with quartz crystal (model no.: 12A4D, Hindhivac make) attached with thermal evaporation unit. After evaporation of Pt metal the power source is switched OFF and the chamber is allowed to cool. The wafers were left in the chamber for half an hour and finally the coated wafers are unloaded from the vacuum coating unit.

3.4.6 Aluminium Metal Deposition on the Back Side of Silicon Wafer

Aluminium metallization has been carried out on the back side of the wafer for making ohmic contact. The vacuum coating unit illustrated in Plate 3.1 was used for metallization. Silicon substrates with thermally grown SiO_2 are placed in the chamber of coating unit in such a way that the back side surface faces the filament and polished surface upward. About 0.5 gm of Al metal is kept in the helical tungsten filament and chamber is closed. Al metallization is done by the resistive heating in the vacuum

coating unit repeating the same process described for platinum metallization in previous section.

3.4.7 Photolithography to Form Gridded Gate Structure

The front side of the wafer was spin coated with negative photoresist using the spinner and then the wafer was prebaked for 10 minutes at 90 ^{o}C. After prebaking, a standard mask of required gridded gate structure was kept on the upper surface of silicon wafer then the surfaces (coated with photoresist) were exposed to UV light for 2 minutes which hardens the photo resist under the transparent part of the mask. Then the photoresist was developed in the developer and unexposed photoresist was dissolved. Now, the wafers were post baked in the oven for about 20 minutes at 150 ^{0}C. The complete lithographic process was carried out in the yellow room. A standard mask which is used for gridded gate structure is shown in Fig. 3.4. The outer and inner diameters of gate structure were kept 1mm and 0.2 mm, respectively. Aqua regia (HNO_3+HCL = 1:3) is used for Pt metal etching. Subsequently, the photoresist from the front side was removed by chemical etching (using the solution consisting of Acetic Acid+TCE + Acetone : 5:20:10).

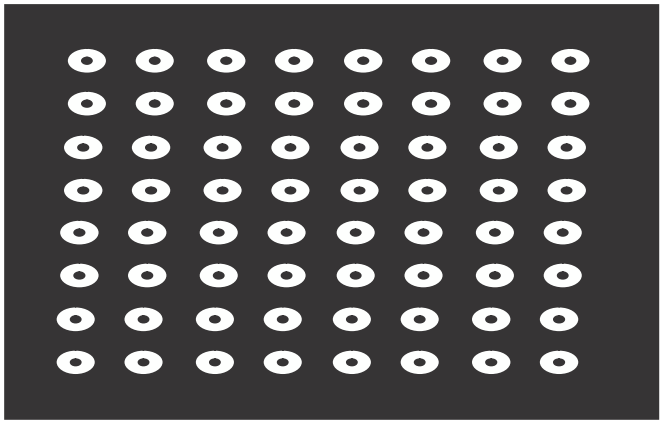

Fig. 3.4 Standard Mask for gridded gate structure

3.4.8 Post Metallization Annealing (PMA)

To get the proper contact between Pt-SiO_2 and Al-Si substrate post metallization annealing is done at 450 ^oC in N_2 ambient for 5 minutes. The annealing has been done in annealing furnace which is shown in Plate 3.8.

Plate 3.8 Annealing furnace

A 3-D Structure of fabricated gridded gate Pt/SiO_2/Si MOS capacitor sensor has been shown in Fig. 3.5.

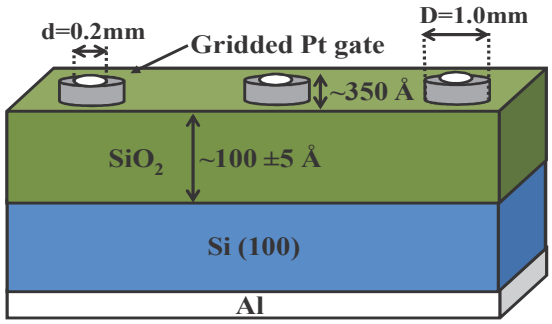

Fig. 3.5 3-D structure of gridded gate MOS capacitor sensor