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Title: Silicon/Porous Silicon composite membrane for high sensitivity pressure sensor

PI: Enakshi Bhattacharya, Professor

Address: Microelectronics and MEMS Laboratory
Electrical Engineering Department,
Indian Institute of Technology Madras
Chennai-600036
India

Phone numbers: 91-44-22574419 (off)

91-44-22576419 (res)

FAX: 91-44-22574402

91-44-22570120

email : enakshi@iitm.ac.in , enakshi.bhattacharya@gmail.com

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Abstract

This project investigates the application of porous silicon (PS) in microelectromechanical systems (MEMS) pressure sensors. The low Young's modulus of PS has been utilized to obtain higher sensitivity in pressure sensors with Si/PS composite membranes. Simulation using Coventorware representing the Si/PS composite membrane with a simple two layer model shows an increase in sensitivity with increase in porosity and thickness of the PS layer. Si/PS composite membranes have been realized by converting a part of the silicon membrane into PS by electrochemical etching of silicon. In composite membranes, the effective Young's modulus reduces which gives more deformation with the application of pressure and hence the sensitivity is higher than that of single crystalline silicon membrane. Pressure sensors with composite membranes have been fabricated and the sensitivity is found to be higher than that of single crystalline silicon membrane increasing with increase in the porosity and the thickness of the PS layer. Composite membranes with microPS and macroPS have been fabricated and their performance has been compared to study the effects of pore diameter and the random/regular pore structure. For the same porosity, sensors with Si/microPS composite membranes show higher sensitivity than Si/macroPS. Both behave linearly at low pressures but the linear range reduces with increase in porosity. For the same porosity, the linear range is more in the case of Si/macroPS than Si/microPS. The composite membranes show irreversible deformation at high pressures, unlike single crystalline silicon membranes. Composite membranes also exhibit higher offset voltage than single crystal silicon membranes. This is found to be caused by the stress developed in the membrane during PS formation and subsequent processing. Use of porous polysilicon piezoresistors to improve the sensitivity has also been reported. The devices have been packaged into TO 39 headers and tested under varying temperature and humidity. The effect of temperature on composite membranes is similar to that on single crystalline silicon membrane. In composite membranes, exposure to humidity reduces the sensitivity of the devices at wafer level. But there is no effect of humidity on packaged devices. We conclude that the high sensitivity and reproducible linear behavior at low pressures make composite membranes a viable option for low pressure measurement. To strike a balance between sensitivity and offset voltage, macroPS may be a better option than microPS.

TABLE OF CONTENTS		Page
ABSTRACT		3
TABLE OF CONTENTS.....		4-5
CHAPTER 1 INTRODUCTION		6-7
1.1	Porous Silicon	6
1.2	Motivation.....	6
1.3	Objectives.....	7
CHAPTER 2 POROUS SILICON		8-16
2.1	Formation of MicroPS.....	8
2.2	Formation of MicroPS.....	8
2.2.1	MacroPS on n-type silicon.....	9
2.2.2	MacroPS on p-type silicon.....	9
2.3	Porosity and thickness Measurement.....	10
2.4	Measurement of Pore Diameter.....	11
2.5	PS morphology with surface profiler.....	11
2.6	Measurement of Young's Modulus by Nano-indentation.....	13
2.7	Optimization of PS for Pressure sensors.....	15
2.8	Conclusion.....	15
CHAPTER 3 SILICON/POROUS SILICON COMPOSITE MEMBRANE PRESSURE SENSORS		17-23
3.1	Simulation	17
3.2	Fabrication of Pressure sensor with Si/Porous silicon composite membrane	18
3.3	Wafer level characterization.....	20
3.3.1	Sensitivity.....	20
3.3.2	Linearity testing.....	21
3.3.3	Deformation of the membrane at high pressures.....	21
3.3.4	Stress measurement.....	21
3.4	Packaging of pressure sensors.....	22

3.5	Conclusion.....	23
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CHAPTER 4	RESULTS AND DISCUSSION	24-37
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4.1	Pressure sensors with Silicon/MicroPS composite membranes	24
4.1.1	Dependence of sensitivity on porosity and thickness of PS	24
4.1.2	Comparison of measured sensitivity with simulated values.....	25
4.2	Performance of Si/MicroPS and Si/MacroPS membranes.....	26
4.2.1	Sensitivity	26
4.2.2	Linearity test.....	27
4.2.3	Dependence of Offset Voltage on porosity.....	27
4.2.4.	Deformation at High Pressures.....	28
4.3	Behavior of Composite Membranes with Self Assembled Monolayer (SAM)	29
4.4	Stress measurement.....	30
4.5	Modified simulation with internal stress.....	31
4.6	Temperature effects on composite membrane.....	33
4.7	Humidity effects on composite membrane.....	34
4.8	Porous silicon piezoresistors.....	34
4.8.1	Critical issue – Ohmic contacts on PPS piezoresistors	35
4.8.2	Testing on various types of pressure sensors	36
4.8.2	Conclusion.....	37

CHAPTER 5	SUMMARY	38-42
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5.1	Summary.....	38
5.1.1	Porous silicon formation.....	38
5.1.2	Pressure sensors with Si/PS composite membranes.....	38
5.1.3	Porous polysilicon piezoresistors.....	40
5.2	Conclusion.....	41

References	43
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1. INTRODUCTION

The many interesting and unique properties of porous silicon (PS) make it a viable material in the field of optics, chemical sensors and microelectromechanical systems (MEMS). The possibility of tailoring the porous structure of PS, as required, leads to many new applications. In this project, we focus on the mechanical and piezoelectric properties of PS to improve the sensitivity of MEMS pressure sensors. This chapter discusses the motivation and objectives of the present work.

1.1. POROUS SILICON

Porous silicon (PS) is generally formed by electrochemical etching of silicon in HF based electrolyte and consists of silicon filaments and voids. It is not a new material and was discovered in 1956 by Ulhir at Bell Labs, USA while he was working on electropolishing of silicon wafers with HF [1]. Porous Silicon (PS) has been used for decades in the fabrication of micromechanical devices. However, it has been mostly used as the sacrificial layer during the fabrication of MEMS devices so far [2]. New applications of PS are possible with an improved understanding of pore initiation and formation process, which enables us to tailor the porous structure according to its application [3].

1.2. MOTIVATION

The motivation of this work is to improve the sensitivity of the MEMS pressure sensor. Increasing sensitivity by using thin silicon membranes suffers from non-linearity under high pressure, known as ballooning effect [4]. Also, thin membranes are difficult to handle. The sensitivity can be improved either by using a material with low Young's modulus for the membrane or a material with high piezoresistive co-efficient for the piezoresistors. The proposed material should have these properties and should also be compatible for integrating with other processes on silicon wafer.

The fabrication of silicone rubber membranes for making microvalves has been reported [5]. The Young's Modulus of silicone is very low (<1 MPa) and so has more deformation. But its maximum operating temperature is 200°C , which will not be suitable for further silicon processing to integrate the system with the sensor.

PS has a very low Young's Modulus and by controlling the formation parameters, the Young's Modulus can be varied. PS is also compatible with silicon IC processing because it is a derivative of silicon. Hence PS is the material of choice for use in the fabrication of pressure sensors to obtain higher sensitivity.

1.3. OBJECTIVES

The main objective of this project is to exploit the unique properties of Porous Silicon (PS) in improving the sensitivity of Silicon based piezoresistive MEMS pressure sensors.

This has been achieved in two ways:

- i. Using a composite Si/PS membrane in MEMS pressure sensors to improve the sensitivity. Young's Modulus of PS depends on the porosity and can be orders of magnitude lower than Si. For the same pressure, this leads to greater deflection for a composite membrane as compared to a Si membrane of the same thickness.
- ii. Replacing the polysilicon piezoresistors with Porous Polysilicon (PPS) since PS has been reported to demonstrate higher piezoresistivity.

In this chapter, we have discussed the applications of PS in MEMS devices, the motivation and objectives of the present work.

2. POROUS SILICON

Porous Silicon (PS) can be formed by wet electrochemical etching or by vapor etching methods. In electrochemical etching, we can have a good control over the properties of PS. The pore geometry and morphology of PS can be varied by controlling the formation parameters such as current density, HF concentration, anodisation time, type and doping level of substrate and illumination state. Its physical, chemical and mechanical characteristics can be tailored according to the specific application or device structure. Based on the geometry of pores, PS is classified into three types according to IUPAC standards as follows [6]:

- (i) MicroPS – average pore diameter less than 10 nm
- (ii) MesoPS – average pore diameter between 10 nm and 50 nm
- (iii) MacroPS – average pore diameter more than 50 nm

2.1. FORMATION OF MICROPS

MicroPS with average pore diameters of few nm can be easily formed on moderately doped p-type substrate (10^{15} - 10^{18} cm⁻³) with aqueous electrolyte in the dark. Figure 2.1 shows the set-up used for porous silicon formation on p-type silicon substrate [7].

The substrates used in our experiments are p-type (100) with resistivity of 1-10 Ω -cm. The cell used to perform electrochemical etching is made of Teflon, which is HF resistant. The silicon wafer itself is the anode and platinum mesh is used as the cathode. The electrolyte is the HF based solution. In our experiments, Iso-propane-alcohol (IPA) is used along with HF to increase the wettability of the silicon surface and to remove the bubbles formed during the reaction. Aluminium metallization is done at the back of the silicon wafer to get ohmic contact. Constant current is passed during the electrochemical etching to have better control over pore formation and to get better reproducibility. The electrochemically-etched surfaces were rinsed in DI water followed by rinsing in low surface tension liquids such as ethanol and then in pentane so that the PS layer had no cracks and was not peeling off.

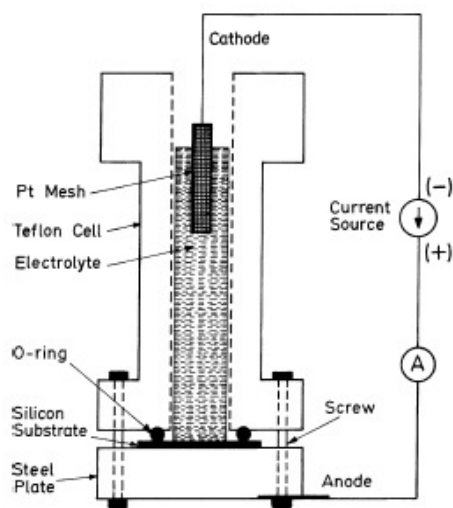


Fig.2.1 Set – up for electrochemical etching of p-type silicon

2.2. FORMATION OF MACROPS

An established method to form MacroPS is by using n-type silicon substrate with aqueous electrolyte [8]. This requires back illumination to generate the holes required for the electrochemical reaction. But recently reports were made on formation of MacroPS on p-type substrate using organic electrolyte [9, 10].

2.2.1. MacroPS on n-type silicon

The most common method of forming MacroPS is using n-type substrate with aqueous electrolyte [11]. The pore diameters in n-type silicon are considerably larger than p-type silicon and form straight channels at low dopant concentrations rather than the randomly directed pores of p-type silicon. Since in n-type silicon, the majority carriers are the electrons, the holes required for electrochemical reaction are supplied by the photogeneration. The back side of the sample is illuminated by light from a halogen lamp as shown in Figure 2.2. The holes generated by the photocurrent diffuse from the bottom of the substrate to the surface and actively participate in the chemical reaction. By varying the intensity of the light, the anodization current can be varied.

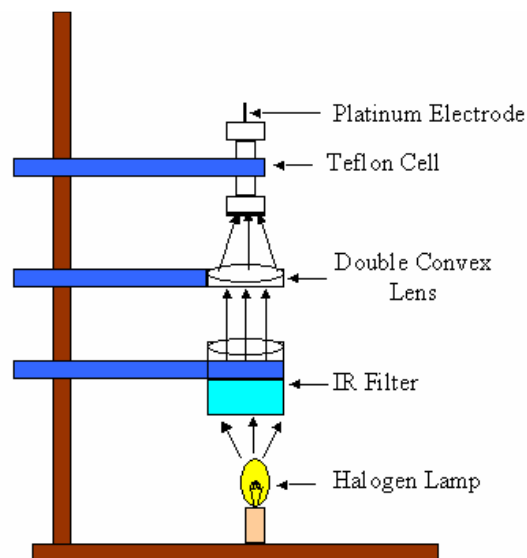


Fig. 2.2 Set – up for electrochemical etching of n-type silicon

It is difficult to get ohmic contact on n-type silicon. Hence phosphorous diffusion has been carried out for n^+ doping at the bottom of the wafer after protecting the top surface by an oxide. Aluminium metal contacts have been made only at the edges so as the light does not get blocked. To allow the light to pass, the stainless steel base has a hole at the center. A double convex lens with a focal length of 5 cm is used to focus the light on to the back of the wafer. The photocurrent is varied by varying the illumination level by an autotransformer. A petridish containing Copper sulphate solution is used to filter the IR to prevent the heating of the sample. A fan is also used to remove the heat on the sample and hence to avoid the thermal generation of carriers.

2.2.2. MacroPS on p-type Silicon

MacroPS on p-type substrate can be obtained with organic electrolytes and this method avoids the complication in the set-up due to back illumination and the additional process steps necessary to get the back ohmic contact. The formation of MacroPS with average pore diameter of around 1 μm on p-type substrate with resistivity of 1-10 ohm-cm using an organic electrolyte containing DMF and HF has been reported [12]. We have followed this procedure in our experiments for formation Macro PS. Measurement of pore diameter and the morphology on surface profiler are discussed in the following sections.

2.3. POROSITY AND THICKNESS MEASUREMENT

Porosity is defined as the fraction of void within the PS layer. The porosity of PS has been determined by gravimetric method [13]. A microbalance (Shimadzu AUW120D) with a resolution (minimum display) of 10 μg has been used. The wafer is weighed before anodisation (m_1), just after anodisation (m_2) and after a rapid dissolution of the whole porous layer in a 3% KOH solution (m_3). The porosity (P) is given by the following equation:

$$P(\%) = \frac{(m_1 - m_2)}{(m_1 - m_3)} \times 100 \quad (2.1)$$

PS layer thickness (d) can be defined by the equation:

$$d = \frac{(m_1 - m_3)}{S_a \rho_d} \quad (2.2)$$

where ρ_d is the silicon density and S_a is the area of etched surface.

2.4. MEASUREMENT OF PORE DIAMETER

The measurement of pore diameter has been done by analyzing the SEM pictures. Figure 2.3 shows the SEM pictures of (a) MicroPS (b) n-type MacroPS and (c) p-type MacroPS. SEM pictures show that MicroPS is random in nature and the pores overlap one another. The average pore diameters in MicroPS are in the order of few nm. The pore diameters measured from the SEM pictures for various formation parameters are given in Table 2.1.

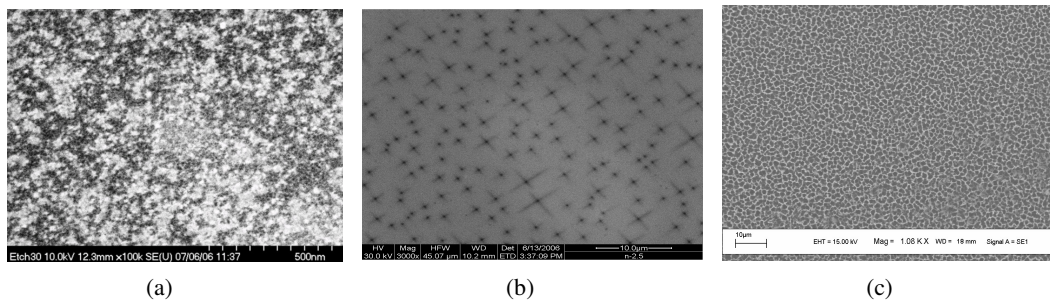


Fig.2.3 SEM pictures of PS (a) MicroPS on p-silicon (b) MacroPS on n-silicon and (c) MacroPS on p-silicon

Table 2.1 Pore diameter measured on PS formed with p-type substrate and aqueous electrolyte

Sample No.	HF (%)	Time (min.)	Current Density mA/cm ²	Porosity (%)	Thickness (μm)	Pore Dia (nm)
1	33	10	10	85	5	9.88
2	20	10	3	85	1	7.93
3	50	10	10	60	5	5.04

The pores of n-type MacroPS are very uniform and the pore diameters are in the order of a fraction of μm. The resultant pore diameter for various formation parameters are given in Table 2.2.

Table 2.2 Pore diameter measured on PS formed on n-type substrate with aqueous electrolyte

Sample No.	HF (%)	Time (min)	Current Density mA/cm ²	Pore Dia (μm)
1	33	10	2.5	0.198
2	33	10	3.5	0.228
3	33	10	5	0.287

Uniform pores equivalent to that of n-MacroPS have been obtained on p-type substrates with organic electrolyte (HF + DMF). The pore diameters of p-type MacroPS formed with organic electrolyte are given in Table 2.3.

Table 2.3. Pore diameter measured on PS formed on p-type substrate with organic electrolyte

Sample No.	HF: DMF	Time (min.)	Current density (mA/cm ²)	Porosity (%)	Thickness (μm)	Average pore dia (μm)
1	1:2	10	10	90	4.8	1.16
2	1:1	10	10	85	5.33	1.52

2.5. PS MORPHOLOGY WITH SURFACE PROFILER

Various pore morphologies have been observed by using surface profiler for different formation parameters. Figure 2.4 shows the PS morphology obtained on (a) p-type substrate and (b) n-type substrate with the help of surface profiler. We can see that the pores on p-substrate are merged and random in nature and their pore dimensions show that the material is MicroPS. The morphology on n-substrate shows uniform pores and their pore dimensions show that the material is MacroPS.

Figure 2.5 shows the MacroPS formed on (a) n-substrate (aqueous electrolyte) with back illumination and (b) p-substrate (organic electrolyte) and both look similar in structure.

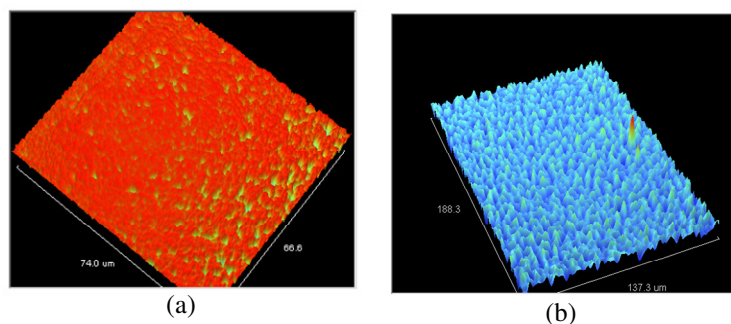


Fig. 2.4 PS formed on (a) p-substrate (aqueous electrolyte) and (b) n-substrate (aqueous electrolyte) with back illumination

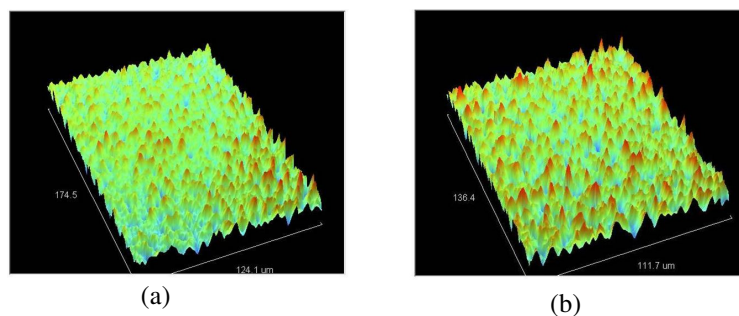


Fig 2.5 MacroPS formed on (a) n-substrate (aqueous electrolyte) and (b) p-substrate (organic electrolyte)

2.6. MEASUREMENT OF YOUNG'S MODULUS BY NANOINDENTATION

Young's Modulus of PS samples has been measured by nanoindentation technique and this technique is easy and non-destructive to the samples. The instruments used for

nanindentation testing were Shimadzu Dynamic Ultra Low Load Microhardness Tester and MTS Nanoindenter XP. The deformation behavior of the film was analyzed via a series of load–displacement ($P-h$) curves.

The measured Young's Modulus of MicroPS formed on p-type (100), 1-10 ohm-cm is 22.11 GPa with 50% porosity reducing to 5.51 GPa for 70% porosity. The Young's Modulus of 70% porosity has been reported as 2.2 GPa [14]. For isotropic PS, the Young's Modulus (E_{ps}) can be written [15] as

$$E_{ps} = E_{Si} (1 - P)^3 \quad (2.3)$$

where P is the porosity and E_{Si} is the Young's Modulus of Silicon.

Table 2.4 shows the measured Young's Modulus of MicroPS, n-type MacroPS and p-type MacroPS with variation in porosity.

Table 2.4 Young's Modulus of MicroPS, n-MacroPS and p-MacroPS samples measured for varying porosity by nanoindentation at IISc, Bangalore.

Type of PS	Porosity (%)	Young's Modulus (GPa)
MicroPS	50	22.11
	70	5.51
n-MacroPS	60	145
	80	85
p-MacroPS	10	25
	70	7

Using Eqn. (2.3), Young's Modulus for 50 % and 70 % PS work out to be 23.75 GPa and 5.13 GPa and are quite close to the measured values given above. The Young's Modulus of n-type MacroPS shows a large variation with porosity, from 145 GPa for 60 % porosity to 85 GPa for 80 % porosity. In p-type MacroPS the Young's Modulus varies within the range of 25 GPa to 7 GPa for the porosity variation of 10 to 70 %. The variation in Young's Modulus with porosity in n-type substrates is larger compared to p-type substrates. Reports and our measurements show the Young's Modulus of PS is much lower than the Young's Modulus of single crystalline silicon and it drastically reduces with increase in porosity.

2.7. OPTIMIZATION OF PS FOR PRESSURE SENSOR

The pressure sensor with Silicon/Porous silicon (Si/PS) composite membrane has been fabricated by converting a part of the silicon membrane into PS by electrochemical etching. The thickness and porosity of the PS layer to be formed on the composite membrane have to be optimized for pressure sensor application.

For comparison of the behavior of composite membranes formed with MicroPS and MacroPS, the total membrane thickness was taken the same as 16 μm with the PS layer thickness being 6 μm and porosities of 50 %, 70 % and 90 %. The formation parameters to get the above thickness and porosity of MicroPS and p-MacroPS are given in Table 2.5.

Table 2.5. Formation conditions for MicroPS and MacroPS for varying porosity with thickness of 6 μm

Type of PS	HF (%)	J (mA/cm ²)	Time (min.)	Porosity (%)	Thickness (μm)
MicroPS (HF + IPA)	80	7.5	12	50	6
	50	5	20	70	6
	33	12	10	90	6
MacroPS (HF + DMF)	80	8.3	9	50	6
	60	5	10	70	6
	50	8.3	15	90	6

2.8. CONCLUSION

In this section, formation of MicroPS, n-MacroPS and p-MacroPS were discussed. SEM pictures and measurement of pore diameter for all the above types of PS under different formation conditions have been discussed. Young's Modulus of MicroPS, p-MacroPS and n-MacroPS measured by nanoindentation technique have been presented and the results show that the Young's Modulus of PS is much lower than that of silicon and it reduces with increase in porosity. The variation in n-MacroPS is higher than the other

two types of PS. The porosity and thickness of PS have been optimized for a total membrane thickness of 16 μm and the required formation parameters have been determined.

3. SILICON/POROUS SILICON COMPOSITE MEMBRANE PRESSURE SENSORS

In this chapter, we discuss about the simulations carried out to explain the behavior of Si/PS composite membranes and the fabrication of Si/PS composite membrane pressure sensors.

3.1. SIMULATION

Simulation was done using Coventorware by representing the Si/PS composite membrane by two layers with different Young's Modulus and Electrothermomechanical analysis was carried out. Total thickness of membrane was taken as 14 μm and size of the membrane as 500 μm x 500 μm and 1000 μm x 500 μm . In the composite membrane structure, the bottom layer is silicon with Young's Modulus of 190 GPa and the top layer is PS with Young's Modulus depending on its porosity. Based on the nanoindentation measurements, the Young's Modulus of PS was taken as 22.11GPa and 5.51GPa for porosity of 50% and 70% respectively. The Young's Modulus for 90% porosity was taken as 0.19 GPa. The simulation was also done for various depths of PS as 2, 4 and 6 (with 90% porosity) out of the total membrane thickness of 14 μm .

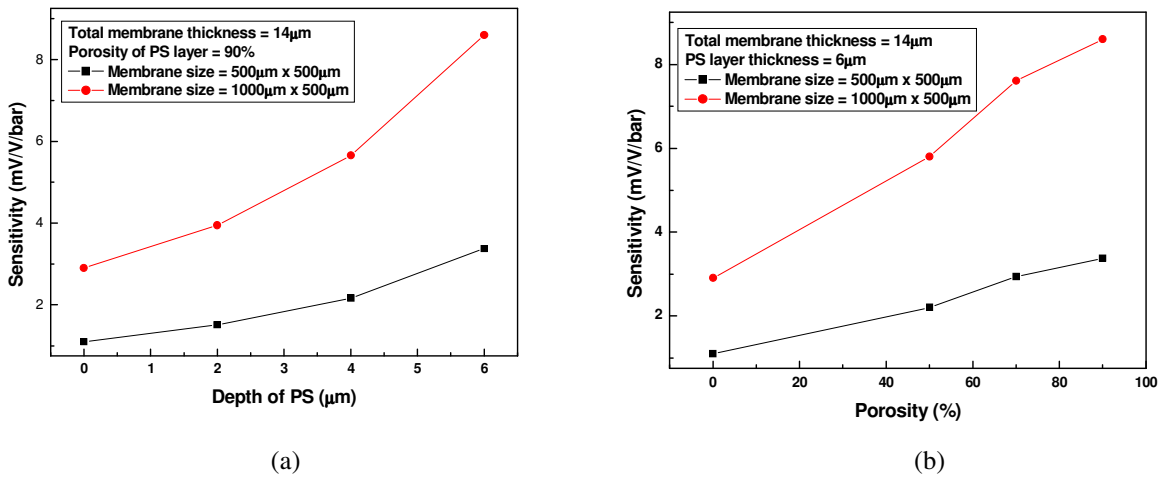


Fig. 3.1 Sensitivity variation obtained on simulated membranes (a) with variation in depth of PS and (b) with increase in porosity of PS

Figure 3.1(a) shows the simulated sensitivity variation for different depths of PS with 90% porosity on composite membrane and the depth of 0 represents the single crystalline silicon membrane. Figure 3.1(b) shows the sensitivity variation for different porosities of PS with 6 μm depth of PS on composite membrane and the 0% porosity corresponds to the single crystalline silicon. The results show the sensitivity increases with increase in depth and also with increase in porosity.

3.2. FABRICATION OF PRESSURE SENSOR WITH Si/PS COMPOSITE MEMBRANE

A p-type silicon <100> double side polished substrate with resistivity of 4-11 ohm-cm, 200 μm thickness was used to fabricate the sensor. The total thickness of the wafer was measured by digital micro screw gauge (Mitutoya). At first a thermal oxide of 0.8 μm was grown by wet oxidation. The topside oxide was then protected by photoresist and the backside oxide was patterned using Mask #1. The silicon was etched from the oxide-patterned portion by anisotropic etching using 30 % KOH at 75°C. The etch rate of this KOH solution is 60 $\mu\text{m}/\text{hour}$. The thickness of the etched silicon was measured with a surface profiler (WYKO NT 1100) and KOH etching was done by controlling etch time till a required membrane thickness was reached. The membrane width was 500 μm and the lengths of the membrane were 500 and 1000 μm . The oxides from both the sides were removed by BHF etching followed by RCA I and RCA II cleaning. Dilute HF dip was given to remove the oxide bonds.

Aluminium metallization was done on the top by thermal evaporation to get the contact for the formation of PS. The wafer was then scribed into individual 2 cm x 2 cm samples to enable loading into the Teflon cell for PS formation. PS was formed on the lower side of the membrane by electrochemical etching for required porosity and thickness. The top metal was removed after the formation of PS. Oxidation of PS is necessary to improve the mechanical stability as well as isolation between the substrate and the polysilicon piezoresistors. A high temperature oxidation will cause the collapse of the PS filaments. Hence, the samples were oxidized at low temperature (450°C-dry) for 30 minutes to avoid collapsing of the PS filaments followed by oxidation at 800°C (dry-wet-dry) for 1-

½ hours. Blue photoluminescence was observed under UV light from the oxidized PS confirming the existence of the porous structure.

A 0.6 µm thick polysilicon layer was deposited using LPCVD at 620°C followed by annealing at 800°C for an hour to relieve any built-in stress in the deposited film. The polysilicon was then doped by diffusion from a Phosphorous source at 800°C for 40 minutes followed by drive-in for 15 minutes. Lithography was done to define the piezoresistors using Mask #2 with IR mask aligner to align the membrane in the bottom to the resistors on the top. The wafer was exposed to HNA solution to etch polysilicon and to form the four resistors. Aluminium metallization was done and patterned using Mask #3. Post metallization annealing was done in Nitrogen ambient at 450°C for 20 minutes for good adhesion. Figure 3.2 shows the mask layout of the pressure sensor and figure 6 shows the cross-section diagram of the sensor with Si/PS composite membrane.

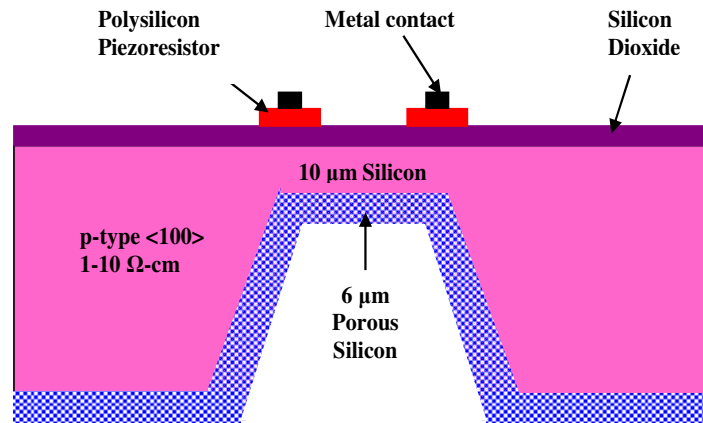


Fig 3.2 Cross section of the fabricated pressure sensor with Si/PS composite membrane

The surface profiler view (using WYKO NT1000) of the device showing the poly piezoresistors connected as Wheatstone bridge is shown in Figure 3.3.

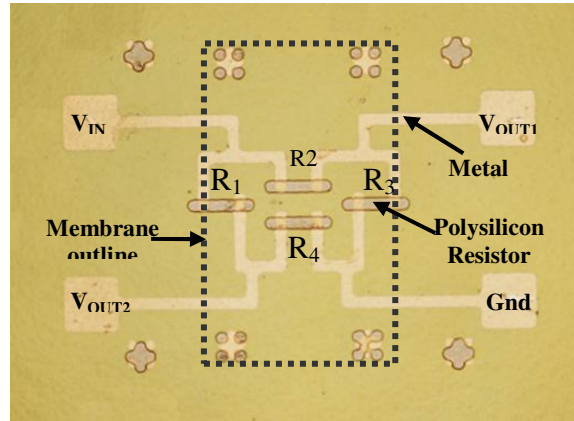


Fig.3.3 Top view of the device showing the poly piezoresistors connected as Wheatstone bridge.

3.3. WAFER LEVEL CHARACTERIZATION

The fabricated devices were tested for measurement of sensitivity, linearity, deformation of the membrane at high pressures, stress on the membrane, temperature and humidity effects. The pore morphology at the bottom of the wafer was observed on the profiler before measurement, which confirmed that the subsequent steps in our fabrication process did not affect the pore structure. The PS surface has a tendency to adsorb moisture from atmosphere. To avoid this, the samples were given a short duration bake (10 min at 80° C) before the sensitivity measurement.

3.3.1. Sensitivity

Testing of the device was carried out at wafer level by placing the sample at the probe station and applying suction at the bottom of the device using a vacuum pump. The input voltage of 1V DC was applied to one of the diagonal arms of the Wheatstone bridge formed by the piezoresistors. The output voltage was measured at the other diagonal arm. Measurement of output voltage was carried out at pressure of 0 and 1 bar by keeping the vacuum pump in the OFF and ON state respectively. The output voltage at 0 bar pressure gives the offset-voltage and the difference of output voltage at 0 bar and 1 bar gives the sensitivity in mV/V/bar.

3.3.2. Linearity Testing

The composite membranes were tested for linearity at pressures less than 1 bar using a vacuum pump with a controlled leak and a manometer. Figure 3.4 shows the set-up used to measure sensitivity at pressures less than 1 bar using a manometer.

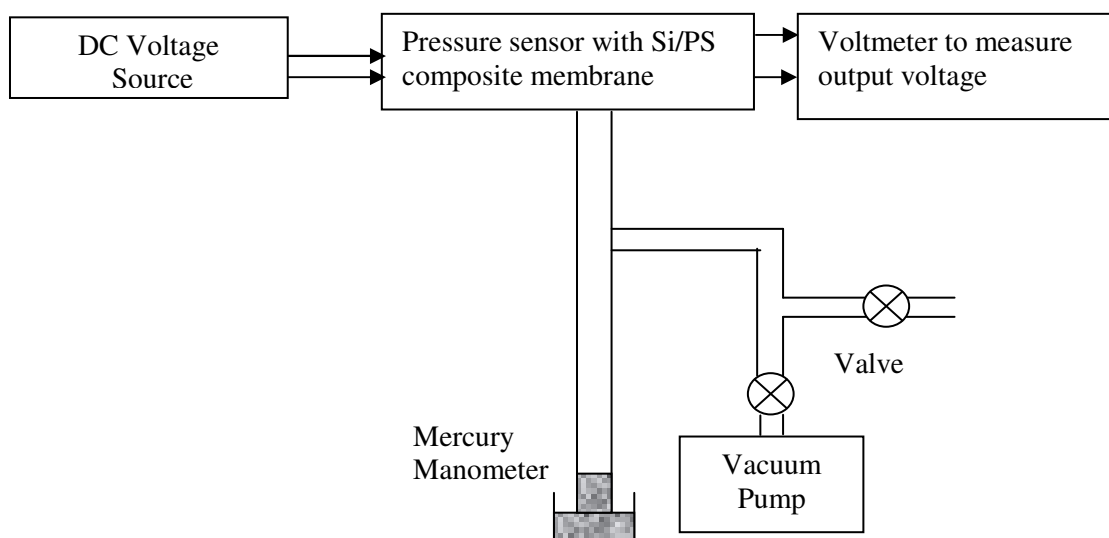


Fig. 3.4 Manometer set-up used for measurement of sensitivity at less than 1 bar

The applied pressure was varied by turning the valve and the pressure was measured from the manometer in mm of mercury. The offset voltage at 0 bar and the output voltages for varying pressures were measured.

3.3.3. Deformation of the Membrane at High Pressures

The membrane was diced out manually and loaded into a jig for application of pressure and the deformation of the membrane was observed on the profiler. Pressures of up to 0.7 bar were applied from a Nitrogen (dry) cylinder and the deformation was observed. The applied pressure was measured by a digital pressure gauge.

3.3.4. Stress Measurement

For the Si/ PS composite membranes, the stress on the membrane can be calculated using Stoney's stress equation [16] (Francia *et al* 2000). Stoney's stress equation is given by equation 3.1 as

$$\sigma = \frac{E_s t_s^2}{6 R_c (1 - \nu_s)^2 t_f} \quad (3.1)$$

where σ is the stress; E_s is the Young's Modulus of the substrate; t_s is the thickness of substrate; t_f is the film thickness; ν_s is the Poisson's ratio of substrate and R_c is the radius of curvature. The radius of curvature of the deformed membrane can be measured by the optical profiler.

3.4. PACKAGING OF PRESSURE SENSORS

To test the effect of temperature and humidity, the fabricated pressure sensor devices have been diced out and packaged into a TO 39 header. A micro drop of adhesive has been taken with a needle and spread evenly with hot air gun on the header. The die has been kept on the header and cured. An efficient sealing has been achieved by using an adhesive which is a combination of LCA -4 (100 parts) and BA-5 (4.5 parts) manufactured by Bacon industries Inc. The curing time was 3 hours at 93°C. Ingress of adhesive into the etched area has to be avoided so as to have effective deflection of the membrane. A hole has been drilled in the header cap for pressure sensing port and then Electron Beam welding has been carried out between header and header cap in vacuum. Typical parameters for E Beam machine are, high voltage 40 kV, Beam current 6 mA and focus current 2.12 A. Thermosonic gold wire bonding has been done to have connection between the device / die pads and the post on the header. Figure 3.5 shows the photograph of the pressure sensor after die bonding and wire bonding.

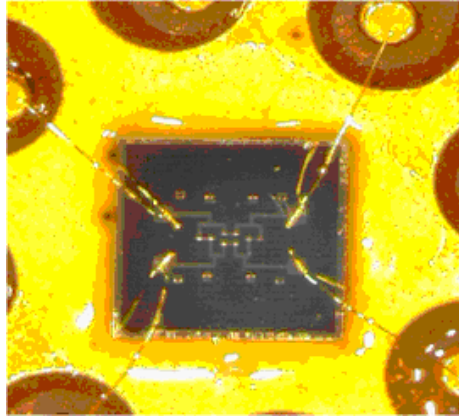


Fig. 3.5 Packaged pressure sensor on a TO39 header showing the gold wire bonding between the metal pads and the posts on the header.

3.5. CONCLUSION

In this section, the optimization of porosity and thickness of PS for use in Si/PS composite membrane pressure sensors were discussed. Simulation of Si/PS composite membrane using Coventorware by a simple two layer structure was discussed. Fabrication details of pressure sensor with Si/PS composite membrane were discussed. Various testing methodologies used to find the performance of Si/PS composite membrane were discussed. Calculation of stress using Stoney's stress equation by measuring the radius of curvature of membrane was discussed. Packaging of pressure sensors to avoid the exposure of PS to ambient conditions was discussed. The results of all the testing procedures mentioned in this section when applied on pressure sensors with silicon membrane and composite membrane will be discussed in the next section.

4. RESULTS AND DISCUSSION

Pressure sensors with Si/PS composite membranes with MicroPS and MacroPS were fabricated as per the process steps mentioned in Sec. 2.1 and 2.2 with the optimized porosity and thickness as mentioned in Sec. 2.7. Testing methods discussed in the earlier chapter were carried out on pressure sensors with Si/MicroPS and Si/MacroPS composite membranes of varying porosity and the obtained results are presented in this chapter.

4.1. PRESSURE SENSORS WITH Si/MICRO PS COMPOSITE MEMBRANES

Pressure sensors with Si/MicroPS composite membranes have been fabricated with PS of (i) varying porosity (50 %, 70% and 90%) with same thickness (6 μm) of PS and (ii) varying thickness (2 μm , 4 μm and 6 μm) with same porosity (90%). The sensitivity at 1 bar pressure were measured and compared to that of a pressure sensor with silicon membrane alone.

4.1.1. Dependence of Sensitivity on Porosity and thickness of PS

The sensitivity plot of the fabricated pressure sensor with PS of 90% porosity with varying depth in the square and rectangular membranes is shown in Figure 4.1. The point corresponding to zero thickness of PS is the single crystalline silicon membrane. Figure 4.2 shows the sensitivity plot of pressure sensor fabricated with PS of 6 μm depth and varying porosities for square and rectangular membranes. The points corresponding to zero percentage porosity is the single crystalline silicon. The results show an increase in sensitivity with increase in porosity and depth of PS formed in the composite membrane.

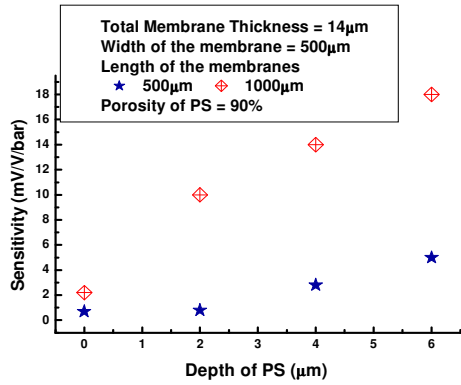


Fig. 4.1 Sensitivity Plot of Pressure Sensor with 90% porosity with varying thickness.

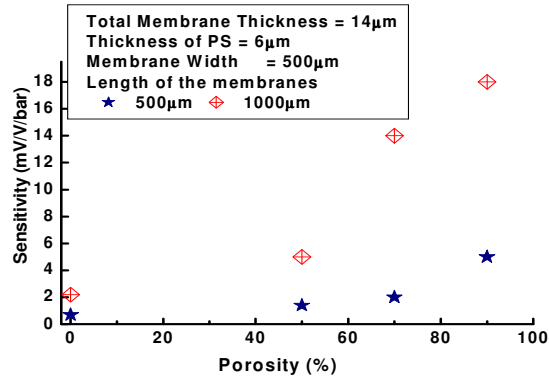


Fig. 4.2 Sensitivity plot of Pressure Sensor with 6 μm PS thickness of varying porosity.

4.1.2. Comparison of measured sensitivity with simulated values

The sensitivity values obtained from the simulations and the fabricated membranes are plotted in Figure 4.3. We can see that the sensitivity of the simulated composite membranes (open symbols) shows the same trend of increase in sensitivity with porosity as seen in the experimental results (solid symbols). But the match is not so good, especially at high porosities. Clearly the simple two layer model is not adequate to explain the behaviour of the composite membranes.

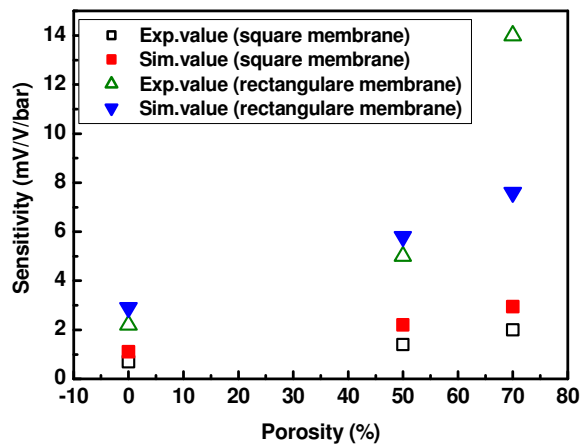


Fig. 4.3 Sensitivity obtained on simulated and fabricated membranes.

4.2. PERFORMANCE OF Si/MICROPS AND Si/MACROPS MEMBRANES

To compare the behavior of composite membranes with MicroPS and MacroPS, the total membrane thickness of 16 μm was fabricated and pressure sensors with MicroPS and MacroPS with thickness of 6 μm were fabricated on this membrane with varying porosity. The results are discussed in the next sub sections.

4.2.1. Sensitivity

Figure 4.4 shows the sensitivity obtained on composite membranes of Si/MicroPS and Si/MacroPS with varying porosity on membrane sizes of 500 μm x 500 μm and 1000 μm x 500 μm . The points corresponding to zero percent porosity represent the sensitivity obtained on membranes of silicon alone. We can see the sensitivity of composite membrane with Si/MicroPS is higher than that of Si/MacroPS. The random structure of pores in MicroPS makes the material spongier than the regular pore structure of MacroPS increasing the deflection. This is also borne out by the nanoindentation measured Young's Modulus on MicroPS and MacroPS with 70 % porosity which were 5.51 GPa and 7.4 GPa respectively. It is also reported in literature that for a given porosity, a MicroPS sample made from p-type silicon is less stiff than a MesoPS sample made from p^+ silicon [14].

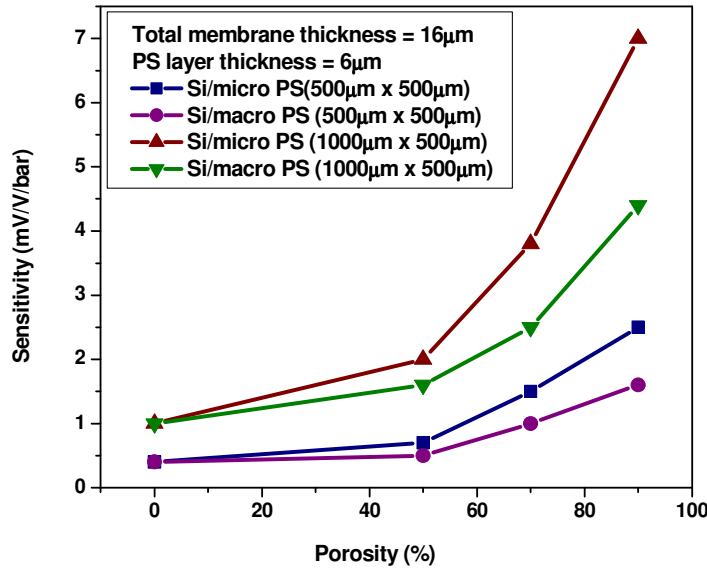


Fig. 4.4 Sensitivity of Composite membranes with Si/MicroPS and Si/MacroPS for varying porosity

4.2.2. Linearity Test

To test the linear behavior of pressure sensors with composite membranes of Si/MicroPS and Si/MacroPS at low pressures, the output voltage of the devices was measured with the pressure varying from 0 to 1 bar with the testing set-up discussed in Sec. 3.3.2. Figure 4.5 shows the behavior of both types of composite membranes of 50% porosity with pressure varying from 0 to 1 bar with offset corrected. We can see that in both Si/MicroPS and Si/MacroPS composite membranes, the output voltages are linear with the applied pressure at pressures less than 1 bar. The sensors were also found to behave linearly at pressures less 1 bar for the other porosities.

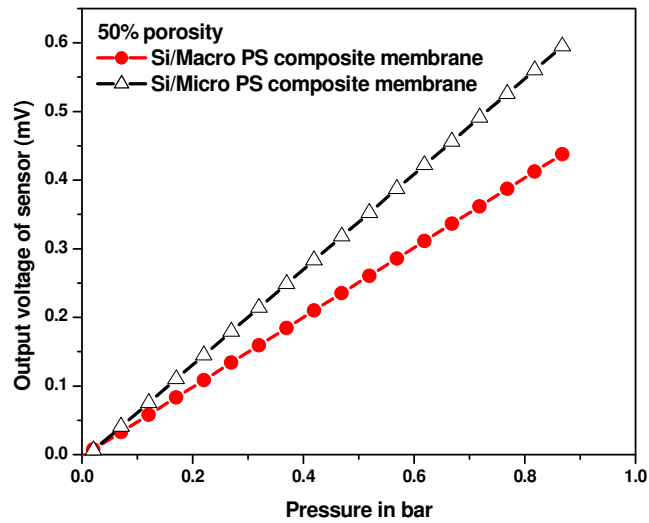


Fig. 4.5 Output voltage of fabricated pressure sensors with composite membranes of Si/MicroPS and Si/MacroPS with offset corrected.

4.2.3. Dependence of Offset Voltage on Porosity

Figure 4.6 shows the offset voltage of pressure sensors with Si/MicroPS and Si/MacroPS measured for varying porosity on a membrane size of $500\text{ }\mu\text{m} \times 500\text{ }\mu\text{m}$. We can see that the offset voltage increases rapidly with the increase in porosity in both types of membranes but, for the same porosity, it is higher in the case of Si/MicroPS than in Si/MacroPS indicating that the formation stress is greater for MicroPS.

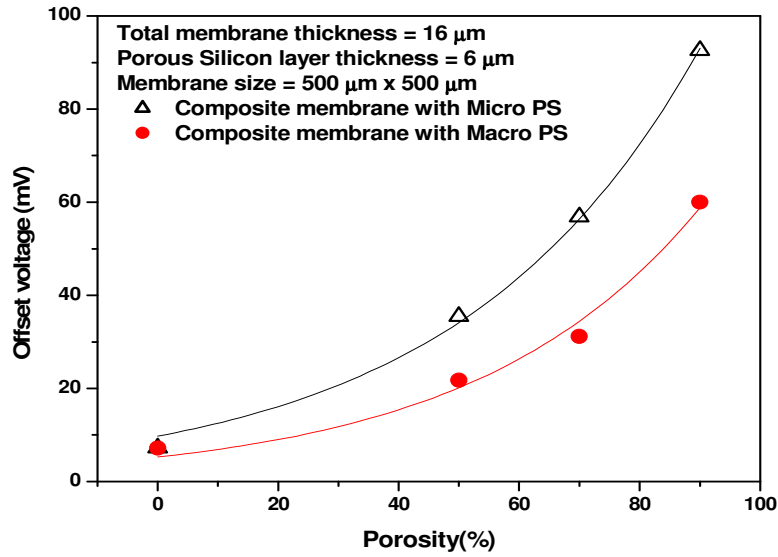


Fig. 4.6 Variation of offset voltage in fabricated pressure sensors with composite membranes of Si/MicroPS and Si/MacroPS with porosity.

4.2.4. Deformation at High Pressures

Figure 4.7 shows the deformations of silicon and composite membranes of Si/MicroPS and Si/MacroPS at high pressures applied from a nitrogen cylinder as mentioned in Sec. 3.3.3. In both types of composite membranes, the deformations are higher than that of silicon membranes and the magnitude of deformation increases with increase in porosity. Both types of composite membranes deform linearly at low pressures, but the linear range reduces with the increase in porosity. The range of linearity is larger in the case of Si/MacroPS than that of Si/MicroPS composite membranes - both showing irreversible deformation at higher pressures. We have seen that for the same porosity, the Young's Modulus of the MacroPS is greater than that of MicroPS. Also, the average pore diameter of MacroPS is three orders higher than that of MicroPS. It is possible that at high pressures, the walls of the pores collapse and come in contact with each other. The resulting surface adhesive forces continue to hold them together even after the removal of the applied pressure. Thus the MacroPS with wider pores can withstand higher applied pressures than the MicroPS samples.

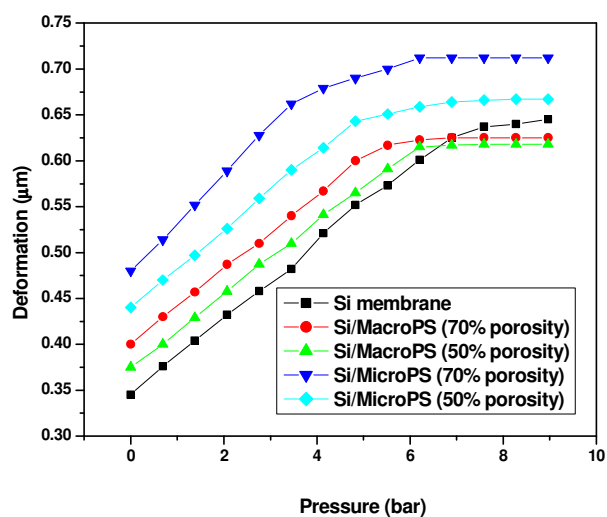


Fig. 4.7 Deformation on silicon and composite membranes at high pressure range.

4.3. BEHAVIOR OF COMPOSITE MEMBRANES WITH SELF ASSEMBLED MONOLAYER (SAM)

The irreversible deformation at high pressures seen in composite membranes can be due to the collapse and adhesion (stiction) of the pore walls. A low surface energy monolayer on the PS membrane can prevent this stiction in the pore walls increasing the linear range. We have used alkanethiol SAMs [19], since it is a simple process not requiring elaborate and expensive equipment or extensive experience to be performed successfully. The monolayer has been formed by immersing the samples in 5 mM solution of 1-Octadecanethiol in ethanol for 24 hours. The devices have been diced out and the deformation of the membrane for pressure up to 10 bar has been observed using an optical profiler. It has been observed that the formation of monolayer on Si/MicroPS composite membrane does not improve the linearity and instead, it deteriorates the reproducibility of the device. But the deformations measured on Si/Macro PS composite membranes with pressure show an improvement in the linear range of operation. Figure 4.8 shows the deformations obtained on Si/Macro PS composite membranes (size of 500 μm x 500 μm) with porosity of 50% and 70% before and after the monolayer formation showing both an enhanced linear range and reversible deformation of the membrane.

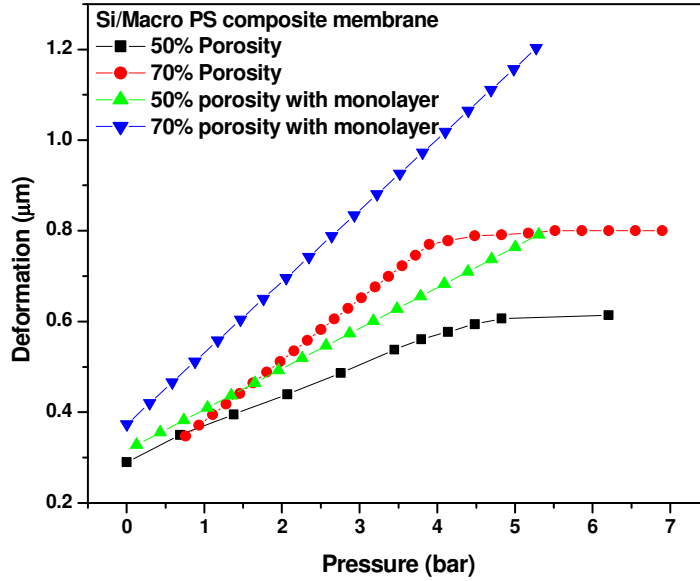


Fig. 4.8 Deformation of Si/Macro PS composite membrane with and without the formation of monolayer.

4.4. STRESS MEASUREMENT

The offset voltage is a result of the residual stress in the composite membranes. The stresses on the composite membranes can be calculated by measuring the radius of curvature on the membrane with surface profiler (WYKO NT1100). Figure 4.9 shows the deformation obtained with surface profiler on composite membrane with Si/MicroPS of 90% porosity. The stress is found to increase with increase in porosity and is higher in Si/microPS composite membranes than Si/macroPS membranes. The fabrication of pressure sensors involves many high temperature steps after the formation of the PS layer. The stress values were also estimated from the deformation of the membrane after completing the processing and are listed in Table 4.1. We see that there is a further increase in stress by a factor of 6-7 from that measured earlier. That the stress indeed plays an important role in the performance of the pressure sensors is seen when it is included in the simulation.

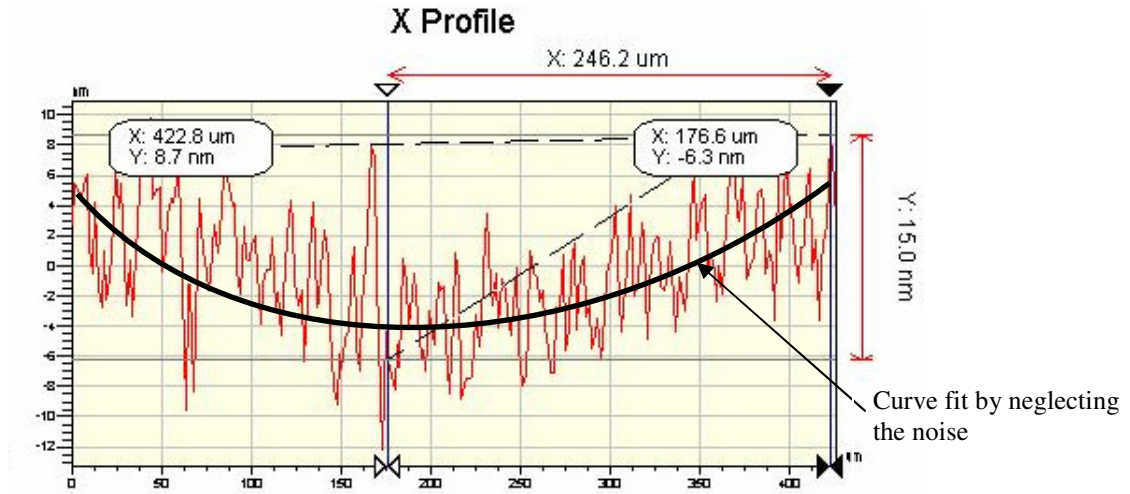


Figure 4.9 Deformation of membrane observed on composite membranes with MicroPS of 90% porosity

Table 4.1 Stress calculated on composite membranes with PS as formed and with finished device

Type of membrane	Porosity (%)	Stress on membrane with as formed PS (MPa)	Stress on membrane after all processing (MPa)
Si/Micro PS	50	0.097	0.69
	70	0.173	1.08
	90	0.378	1.76
Si/MacroPS	50	0.088	0.58
	70	0.146	0.89
	90	0.204	1.43

4.5. MODIFIED SIMULATION WITH INTERNAL STRESS

We had earlier discussed the results of simulation of the behaviors of silicon and composite membranes in Section 4.1.2 using the MEMS simulation software Coventorware. Though the sensitivity obtained by simulation showed a similar trend of increase with porosity as seen in practice, the match was poor with the experimental values. We have done simulations again including the measured stress values on the composite membranes and Figure 4.10 shows the sensitivity obtained from simulation and measurements. The simulated sensitivity results closely match with the experimental results on introducing the measured stress values on simulated

membranes. The sensitivity obtained on simulated membranes is slightly higher than the results obtained on experimental membranes up to 70% porosity. On composite membrane with PS of 90% porosity the sensitivity obtained by simulation is less than the fabricated one. The reason may be the error in the measurement of stress at higher porosity.

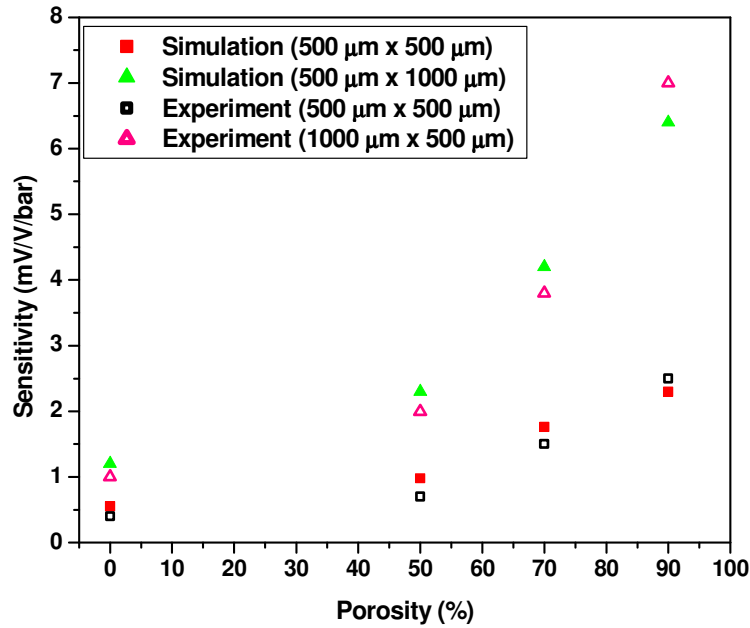


Fig.4.10 Sensitivity obtained on simulated (with internal stress) and fabricated membranes.

Stress on the composite membrane was calculated by Stoney's equation. Stoney's equation has the following assumptions [17]:

- substrate/thin film system is mechanically free
- film thickness (t_f) is much smaller than substrate thickness (t_s)

However, in our case, the above assumptions are not valid as the membrane is fixed on all the edges to the wafer and the ratio of film thickness (t_f) to substrate thickness (t_s) is 0.6. Hence, the mismatch in sensitivity of simulated and fabricated membranes may be attributed to the non-adherence of the above mentioned assumptions. Measurement of stress has to be done by using a different technique particularly in composite membranes with PS of 90% porosity.

4.6. TEMPERATURE EFFECTS ON COMPOSITE MEMBRANE

The fabricated pressure sensor devices were diced out and packaged into a TO 39 header. The packaged pressure sensor has been mounted on a specially fabricated jig to apply pressure. A DC input voltage of 1V was given to the input terminals of the Wheatstone bridge on the pressure sensor and the output voltage from the pressure sensor was measured using a digital multimeter. Pressure was applied from a Nitrogen cylinder. The applied pressure was varied using a control valve and measured by using a digital pressure gauge. The packaged sensors were subjected to different temperatures ranging from 30 – 115°C. The measured sensitivity at different temperatures for silicon membrane and Si/PS composite membrane are given in Figure 4.11.

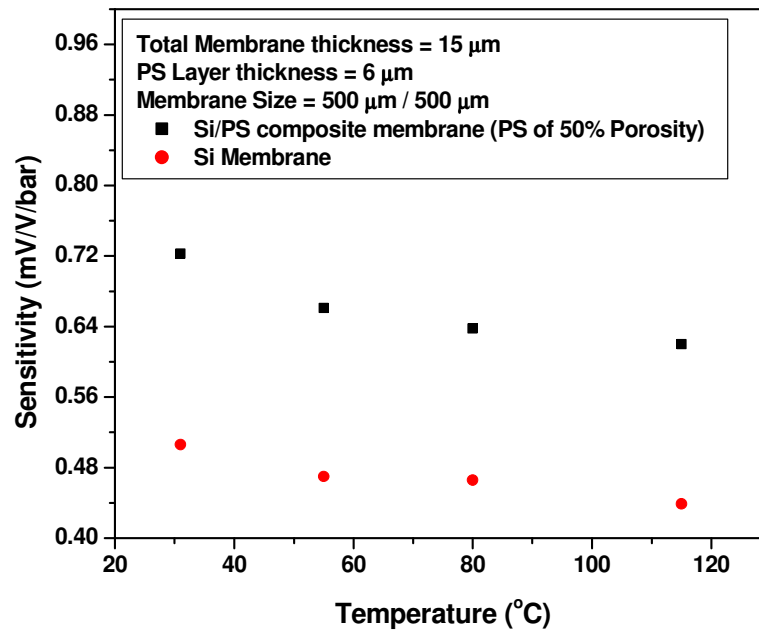


Fig. 4.11 Sensitivity of silicon and Si/PS composite membranes for varying temperatures

At room temperature (30°C), sensitivity of PS/Si membrane is 0.72 mV/V/bar and the sensitivity of silicon membrane is 0.506 mV/V/bar. Sensitivity decreases with temperature in both types of membrane and percentage change in sensitivity due to temperature variation from 30°C to 115°C is 14.25% for Si/PS composite membrane sensor and is 13.25% for Si membrane sensor. Hence the effect of temperature on

composite membrane is almost the same as that of silicon membrane and it is due to the change in resistance of polysilicon piezoresistors with temperature.

4.7. HUMIDITY EFFECTS ON COMPOSITE MEMBRANE

Porous Silicon is very sensitive to humidity due to its high surface to volume ratio leading to greater adsorption [18]. To test the effects of humidity on composite membranes, both unpacked wafer level and packaged sensors with Si/PS composite membrane were kept inside a chamber. Nitrogen gas from cylinder was bubbled through a water bubbler with water boiling at 100°C and passed through the chamber for 90 minutes. Then the samples were taken out and tested for sensitivity and offset voltage and the output voltage at a pressure of 1 bar. The measured sensitivity and offset voltage before and after passing water vapor are given in Table 4.2.

Table 4.2 Offset voltage and sensitivity variation of packaged and unpackaged devices of composite membranes with humidity

Si/PS Composite membrane	Offset voltage (mV)		Sensitivity (mV/V/1 bar)	
	Before applying humidity	After applying humidity	Before applying humidity	After applying humidity
Wafer level	18.64	18.72	0.55	0.49
Packaged device	17.32	17.34	0.71	0.71

We can see that the wafer level (not packaged) devices are sensitive to humidity showing a reduction in the sensitivity. But the packaged device show only slight variation in offset voltage and there is no change in sensitivity. Hence, there is no effect of humidity in packaged device since the porous side is sealed on the die mount with a strong adhesive and is not exposed to water vapor.

4.8. POROUS POLYSILICON PIEZORESISTORS

This section describes the technique of improving the sensitivity of bulk micromachined silicon pressure sensors by using porous polysilicon (PPS) piezoresistors. Further

increase in sensitivity can be obtained by converting the polysilicon piezoresistors on the membrane into porous polysilicon (PPS). Comparison of performance has been made for (i) silicon membrane with polysilicon piezoresistors (ii) silicon membrane with PPS piezoresistors (iii) Si/PS composite membrane with polysilicon piezoresistors and (iv) Si/PS composite membrane with PPS piezoresistors.

4.8.1. Critical issue – ohmic contacts on PPS piezoresistors

Making good ohmic contact to porous silicon is very difficult due to surface roughness. Hence in this work we convert the polysilicon into porous polysilicon after making the contacts. In the fabrication of pressure sensors, after the polysilicon is patterned on the membrane, aluminium is evaporated and patterned. Then the sample is exposed to HNA vapor to form PPS by protecting the metal by photoresist. Since the metal contacts are taken from polysilicon, the contacts are good. The resistance measured between the diagonal arms of the Wheatstone bridge after formation of PPS is higher than the resistance measured before the polysilicon is converted into porous.

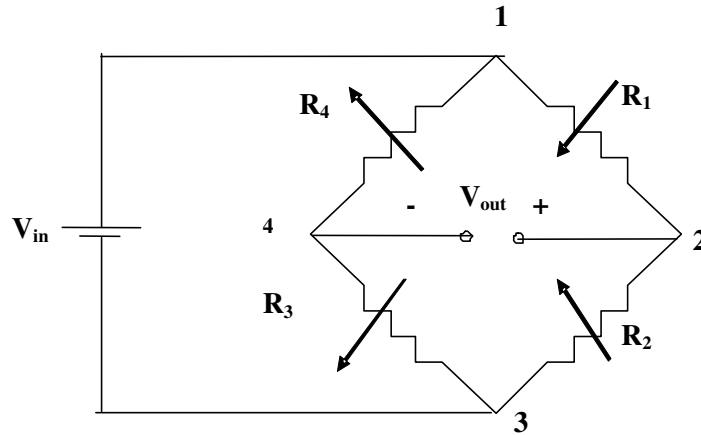


Fig. 4.12 Wheatstone bridge with the nodes for measurement

Table 4.3 shows the resistances measured between the diagonal arms of the Wheatstone bridge on $500\mu\text{m} \times 500\mu\text{m}$ and $1000\mu\text{m} \times 500\mu\text{m}$ silicon membranes with polysilicon piezoresistors and PPS piezoresistors. R_{13} is the resistance between the nodes 1 and 3 and R_{24} is the resistance between the nodes 2 and 4 as shown in Figure 4.12. Row 2 shows the resistance is higher in sensors with PPS piezoresistors than sensors with polysilicon piezoresistors as expected.

Table 4.3 Measured resistance between diagonal arms of Wheatstone bridge

Sample No.	Type of membrane / Type of Piezoresistor	Membrane size (500 μ m x 500 μ m)		Membrane size (1000 μ m x 500 μ m)	
		R ₁₃ (k Ω)	R ₂₄ (k Ω)	R ₁₃ (k Ω)	R ₂₄ (k Ω)
1	Si membrane / Polysilicon piezores.	1.44	1.47	1.42	1.47
2	Si membrane/ PPS piezores.	2.51	2.57	2.50	2.61

4.8.2. Testing on various types of Pressure Sensors

Four different types of pressure sensors were fabricated. (i) silicon membrane with polysilicon piezoresistors (ii) silicon membrane with PPS piezoresistors (iii) Si/PS composite membrane with polysilicon piezoresistors and (iv) Si/PS composite membrane with PPS piezoresistors. The composite membranes were made with MicroPS of 50% porosity with depth of 6 μ m. PPS were formed with the optimized HF concentration and exposure time. The testing was done on membrane sizes of 500 μ m x 500 μ m and 1000 μ m x 500 μ m with thickness of 16 μ m. The sensors were tested at wafer level with the applied pressure of 1 bar and input voltage of 1 V DC.

Table 4.4 Sensitivity and offset voltage of various types of pressure sensors

Sample No.	Type of membrane / Type of Piezoresistor	Sensitivity (mV/V/bar)		Offset voltage (mV)	
		(500 μ m x 500 μ m)	(1000 μ m x 500 μ m)	(500 μ m x 500 μ m)	(1000 μ m x 500 μ m)
1	Si membrane / Polysilicon piezoresistors	0.4	1	7.2	8
2	Si membrane/ PPS piezoresistors.	0.5	1.5	8	10
3	Composite membrane/ Polysilicon piezoresistors.	0.7	2	35.5	65
4	Composite membrane/ PPS piezoresistors.	1.4	4	42	69

4.9. CONCLUSION

Performance of pressure sensors with Si/MicroPS and Si/MacroPS composite membranes in terms of their sensitivity, deformation at high pressures, offset voltage for varying porosity and the stress on the membranes have been discussed. The results of simulation of composite membranes with internal stress on the membrane have been reported. Finally the devices with composite membranes have been tested for temperature and humidity effects. In our wafer level measurements, moisture was removed by baking. Once the samples are packaged such that the PS side is sealed, then the ambient conditions do not affect the device performance. We found the error in measurement of sensitivity and offset voltage are less than 2 % and 5 % respectively in our measurements. Besides normal process variation (lithographic, temperature gradient in diffusion furnace) another contribution to the error is due to variation in uniformity of PS formation. The improvement in sensitivity of pressure sensors with PPS piezoresistors was presented. The formation of PPS by vapor etching for 5 sec does not introduce any adverse effect on stress and, as shown by the symmetry of the resistance arms, is found to be similar to pressure sensor with silicon membrane with polysilicon piezoresistors.

5. SUMMARY AND CONCLUSION

This section summarizes the work carried out and gives the conclusion about the PS formation, fabrication of pressure sensors with Si/PS composite membrane and the improvement in sensitivity by using PPS piezoresistors.

5.1. SUMMARY

5.1.1. Porous Silicon Formation

Porous Silicon was formed by electrochemical etching of silicon for various formation parameters. The porosity and thickness of PS layer were measured by gravimetric method. MicroPS was formed on (100) p-substrate of 1-10 Ω -cm resistivity with aqueous (HF + IPA) electrolyte. MacroPS was formed on (100) n-substrate of 1-10 Ω -cm resistivity with aqueous (HF + IPA) electrolyte in a special set-up with provision for back illumination. MacroPS formation on p-substrate with organic electrolyte was also discussed. This has the dual advantages, as compared to formation of MacroPS on n-substrate, that there is no requirement for back illumination and the formation of n^+ back contact making the process much simpler. SEM measurements were carried out to measure the pore diameter. The porosity and thickness were measured by gravimetric method. Nanoindentation technique was used to measure the Young's Modulus of PS.

5.1.2. Pressure Sensors with Si/PS Composite Membranes

Behavior of composite membrane pressure sensors was simulated in Coventorware using a simple two layer model. The porosity and thickness of PS layer were optimized for pressure sensor application with the idea of converting a part of the total silicon membrane layer to a certain depth into PS. Pressure sensors with Si/MicroPS and Si/MacroPS on same total membrane thickness of 16 μ m with PS layer thickness of 6 μ m were fabricated. Samples were prepared with PS of varying porosity. Testing of devices was carried out at wafer level on a probe station at a pressure of 1 bar using a vacuum pump. Devices were tested for linearity at pressures less than 1 bar using a home built set-up with a manometer and a vacuum pump with a controlled leak. Deformations of composite membranes at higher pressures were measured with the surface profiler. A higher offset voltage on composite membranes and its increase with increase in porosity was observed. Behaviour of composite membranes with self-assembled monolayer was

studied. Stress measurements were carried out on composite membranes of Si/MicroPS and Si/MacroPS for varying porosity on membranes with (i) as formed PS and (ii) after all process steps were completed. Temperature and humidity effects on composite membranes were tested after the devices were packaged into TO 39 headers.

In our study, we exploit the mechanical properties of PS using it as part of the membrane. As the porosity increases, the Young's Modulus reduces and the deformation and sensitivity values increase. Hence, there is no optimum porosity as such for the composite membrane. However, at higher porosities of PS, due to weakness in the material, the linear range of operation reduces. Thus, there is a trade-off between sensitivity and linear range and the porosity has to be decided by the measurement range of pressure to be applied. At low level of pressure (less than 1 bar), porosity up to 90 % can be used.

As PS is not used as an electronic sensing material, the effect of temperature on piezoresistivity of PS will not affect the sensitivity of the device fabricated in this way. The sensitivity of pressure sensor strongly depends on the membrane dimensions, especially on the membrane thickness. The data sheets of many commercial pressure sensors do not specify the membrane dimensions. Also, the commercial pressure sensors contain integrated amplifier and temperature compensation circuitry. Table 5.1 shows the data collected on some of the commercial pressure sensors and the pressure sensors fabricated in the labs. We can see that the sensitivity of our composite membrane pressure sensors is higher than the others. For Sl. Nos. 9 and 10, the devices have Si/PS sandwich type membrane with variable thickness of PS and the piezoresistance of the membrane is used to measure the sensitivity. This involves making electrical contacts on PS which are not reliable.

Table 5.1 Comparison of sensitivity of various pressure sensors

Sl.No.	Author/Industry	Integrated Amplifier / Temperature compensation circuitry	Sensitivity (mV/V/100 kPa)	Diaphragm thickness (μm)
1	Freescall semiconductor Inc. USA Technical Data (2005)	Yes	400	Not known
2	Endevco Corp. CA Product data (2008)	Yes	30	Not known
3	Vinoth kumar (2006)	Yes	31.6	11
4	Institute for Micro-electronics and Information Technology, Germany, Maxim application Note 871 (2001)	No	2	15
5	Tankiewicz <i>et al.</i> , (2001)	No	3.4	5
6	Yang <i>et al.</i> (2005)	No	13	4
7	Sivakumar (2006)	No	2	15
8	Present work (2008)	No	7	10μm/6 μm Si/PS composite membrane
9	Pramanik and Saha (2006)	No	8	5 μm /95 μm PS/Si
10	Pramanik and Saha (2006)	No	27.5	20 μm /80 μm PS/Si

5.1.3. Porous Polysilicon Piezoresistors

The formation of Porous Polysilicon (PPS) by vapor etching was discussed. The pore size was optimized for the dimensions of the piezoresistors used in the pressure sensors by controlling the HF concentration and the etching time. Critical issue of obtaining ohmic contact on PPS piezoresistors was discussed and the process steps used to get better ohmic contact was also discussed. Pressure sensors with PPS piezoresistors were fabricated and tested. Comparison of behavior of four types of devices – (i) silicon membrane with polysilicon piezoresistors (ii) Si/PS composite membrane with

polysilicon piezoresistors (iii) silicon membrane with PPS piezoresistors and (iv) Si/PS composite membrane with PPS piezoresistors were discussed.

5.2. CONCLUSION

Listed below are the conclusions drawn from the present work:

1. Simulation of pressure sensors with composite membranes shows the trend of increase in sensitivity with increase in porosity and thickness of the PS layer.
2. Pressure sensors fabricated with Si/PS composite membranes show higher sensitivity than that of single crystalline silicon alone with the sensitivity increasing with increase in the porosity and the thickness of the PS layer. Pressure sensors with Si/MicroPS composite membranes show higher sensitivity than Si/MacroPS composite membranes.
3. The composite membranes behave linearly at low pressures and saturate at high pressures. The range of linearity reduces with the increase in porosity. Si/MacroPS membranes show greater range of linearity than Si/MicroPS.
4. Pressure sensors with composite membranes show higher offset voltage than silicon membranes and this is due to the internal stress on the membrane caused by PS formation. Offset voltages and the estimated stress values are found to be higher in Si/MicroPS composite membranes than Si/MacroPS composite membranes.
5. Stress measurement on composite membranes on finished devices show that the stress increases by processes such as oxidation and diffusion following the PS formation. Sensitivity of simulated membranes with internal stress shows a good match with experimental values up to 70% porosity. Simple Stoney's equation is not enough to find the stress on composite membranes as the assumptions of Stoney's equation are violated for composite membranes.
6. The composite membranes e irreversible deformation at high pressures.
7. Use of self-assembled monolayer as anti-stiction coating helps to increase the linear range of operation in Si/MacroPS composite membranes.
8. Sensitivity of pressure sensors are further increased by use of PPS piezoresistors due to improved piezoresistance coefficient.

9. Packaged pressure sensors show that the temperature effects on Si/PS composite membranes are almost the same as in silicon membranes.
10. Sensitivity of sensors reduces on exposure to humidity before packaging since the PS adsorbs moisture but is negligible in packaged sensors.

Finally, we can conclude that pressure sensors with Si/PS composite membrane with offset correction are a viable option for measurement of low pressures due to their high sensitivity and linearity.

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