Detection of Xyelene (C_8H_{10}) by Pd-gate MOS Sensor

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Abstract— *A sensor based on Pd/SiO_2/Si MOS capacitor was fabricated on p type <100> (1-6 ΩCm) Si with thermal oxide layer of thickness about 200 A˚. The sensor showed sensitivity to Xyelene (C_8H_{10}) vapour and was characterized at Xyelene concentrations ranging from (500ppm-16,000ppm) at different operating temperatures (room temperature,70˚C and 120˚C), in air. It was found that sensitivity of the sensor was maximum at an operating temperature of 70˚C.*

Keywords— MOS (metal-oxide- semiconductor) structure, gas sensor, Palladium (Pd), sensitivity, C-V and G-V characteristics.

I. INTRODUCTION

The sensors capable of detecting the toxic as well as explosive gases such as Xyelene which is known to be having very serious effects on the respiratory systems are need to be developed. The promise of small integrated sensors with high sensitivity has motivated the numerous researchers [1-4] to develop and study the gas sensors based on Pd gate MOS structure. These sensors have been found to be sensitive for hydrogen and the detection mechanism could be attributed to change in metal work function on exposure to hydrogen. [5,6]. The model for mechanism of hydrogen sensitivity of Pd MOS device has been proposed by Lundstrom et al. [7]. Exposure of hydrogen gas on the sensor causes the hydrogen molecule to dissociate into atomic hydrogen on Pd surface, and then these atoms diffuse through the Pd film and get adsorbed at Pd/insulator interface. The adsorbed and dissolved hydrogen atoms give rise to the formation of dipole layer at the Pd/ insulator interface and work function of the Pd is decreased which can be measured in terms of change in flat band voltage of the MOS capacitor [7]. It is possible to detect the hydrocarbons or other hydrogen containing gases by these sensors provided they can be dehydrogenated on the Pd surface, so that hydrogen atoms can diffuse and are adsorbed on Pd/ insulator interface and give rise to change in the flat band voltage of the device. This paper presents the result of, analysis of C-V and G-V responses of Pd gate MOS capacitors, exposed to various Concentration (500 ppm – 1. 6 %) of Xyelene vapors, in air at several temperatures. It is found that Pd gate becomes more sensitive to Xyelene, in air at operating temperature 70˚C.

II. EXPERIMENTAL

The SiO_2 based Pd- gate MOS structure was fabricated on a p-type <100>, 3″ Silicon substrate and its structure is shown in fig. 1.

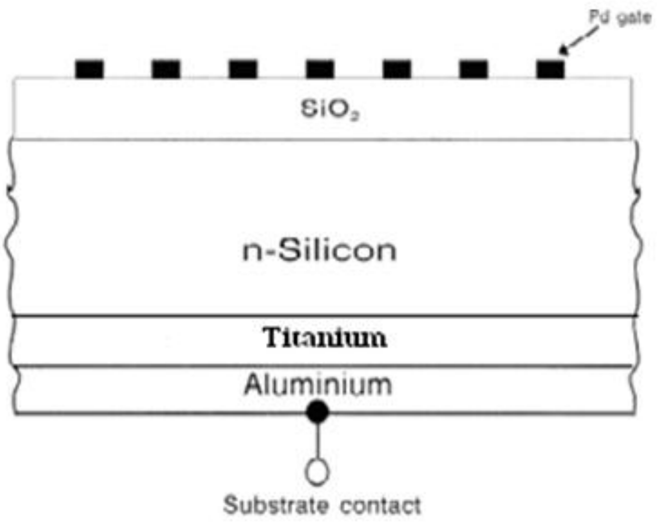

FIGURE 1: Cross sectional view of the fabricated sensor

For fabrication of MOS capacitor, the wafer was thoroughly cleaned using standard technological cleaning procedures used in silicon technology. SiO_2 layer was grown by dry thermal oxidation of silicon wafer in the oxidation furnace (at 900^0C for 12 min.). Subsequently, photolithography technique was used for retaining front side oxide and removing back side oxide.

Palladium was deposited on the front face of the wafer by vacuum evaporation method, using standard mask having holes of 1mm. diameter for gate. The ohmic contact to the back side of Si substrate was made by evaporating Al metal. Annealing was done at 450^0 C in nitrogen ambient for 7 minutes, for achieving a proper front and back contacts. The experimental set–up used to study the C-V and G-V response of the fabricated device with exposure to xyelene is shown in fig 2. C-V and G-V characteristics of fabricated MOS sensor was studied at different concentration of dyelines with the help of C-V analyzer, (model 590 KEITHLEY Instruments, USA) and Precision LCR meter HP-4284A (having frequency range of 20Hz-1MHz). Both the Instruments were interfaced to a PC. ICS (Interactive characterization software) software was used to obtain the accurate information from the instruments and stored in computer.

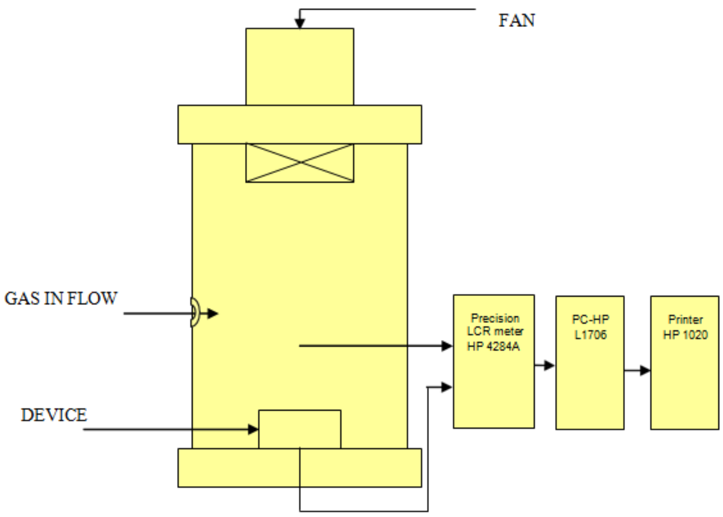

FIGURE 2: Block diagram for experimental set-up

III. RESULTS

3.1 C-V measurements

The variation of capacitance with gate voltage for fabricated Pd gate MOS capacitor, in air, as well as upon exposure to different concentrations (500 ppm- 16,000 ppm) of xyelene at 100 kHz frequency was recorded at different operating temperatures(room temperature, 70˚Cand 120˚C) and the obtained results are shown in fig (3-5).

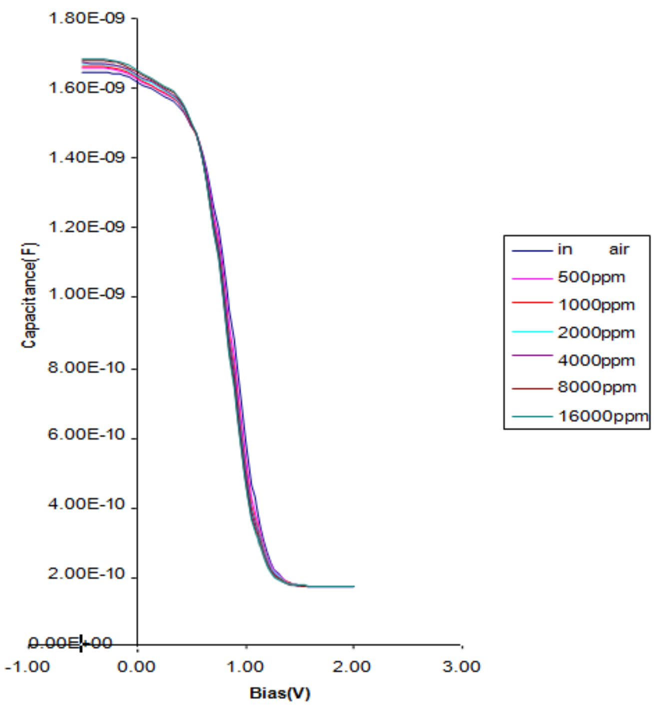

FIGURE 3: C-V response of Pd gate MOS sensor at room temperature

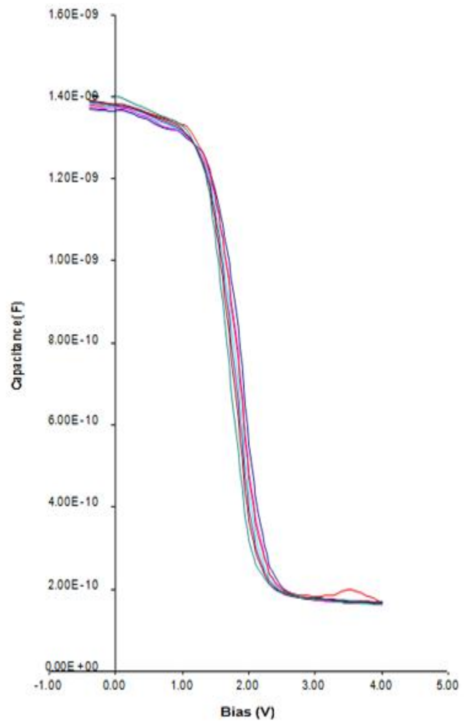

FIGURE 4: C-V response of Pd gate MOS sensor at 70^{0}C

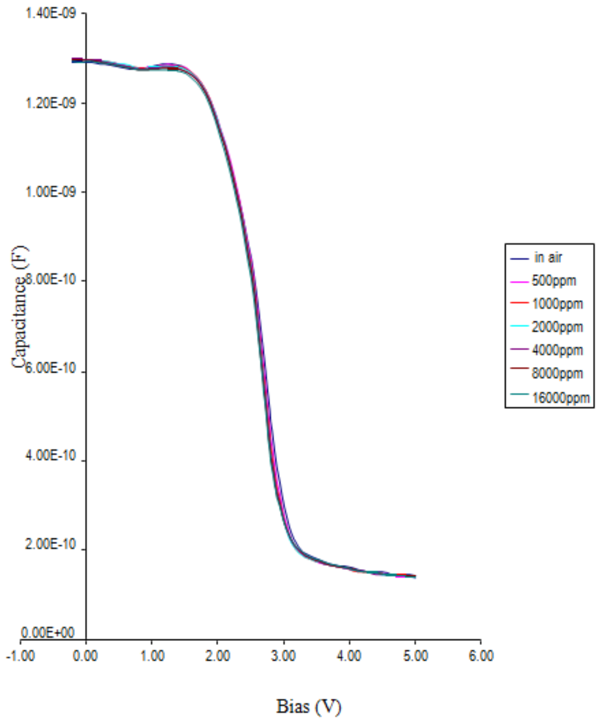

FIGURE 5: C-V response of Pd gate MOS sensor at 120^{0}C

It can be observed that exposure of the sensor to higher Xyelene concentration causes a shift of whole C-V curve to more negative side of the voltage axis and this shift is most prominent when device is heated at temperature at of 70˚C. It is also inferred from these figures that as the concentration of Xyelene increases capacitance decreases and maximum change in the capacitance always occurs at the same bias voltage for all concentration levels at a given operating temperature and sensitivity increases with the higher concentration.

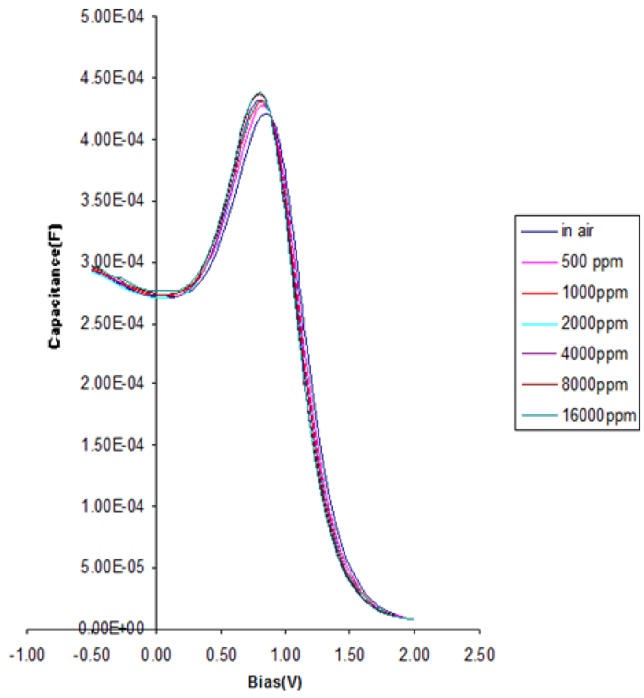

FIGURE 6: G-V response of Pd gate MOS sensor at room temperature

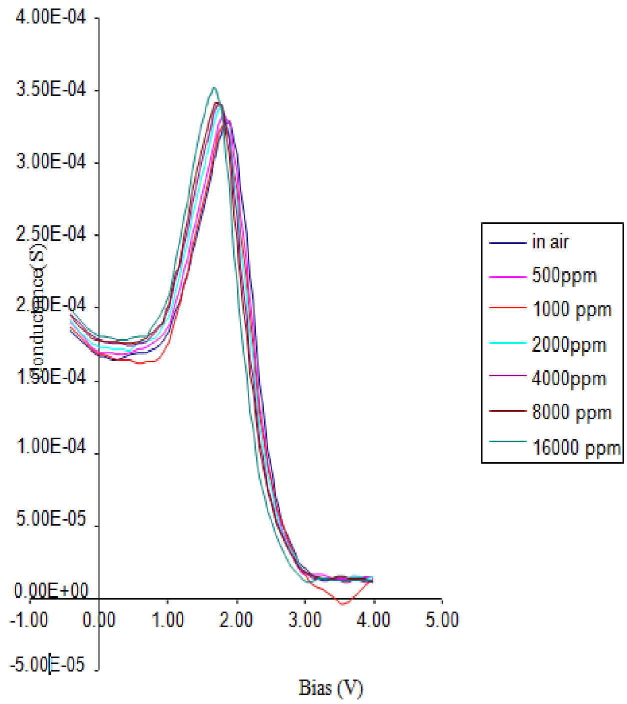

FIGURE 7: G-V response of Pd gate MOS sensor at 70^{0}C

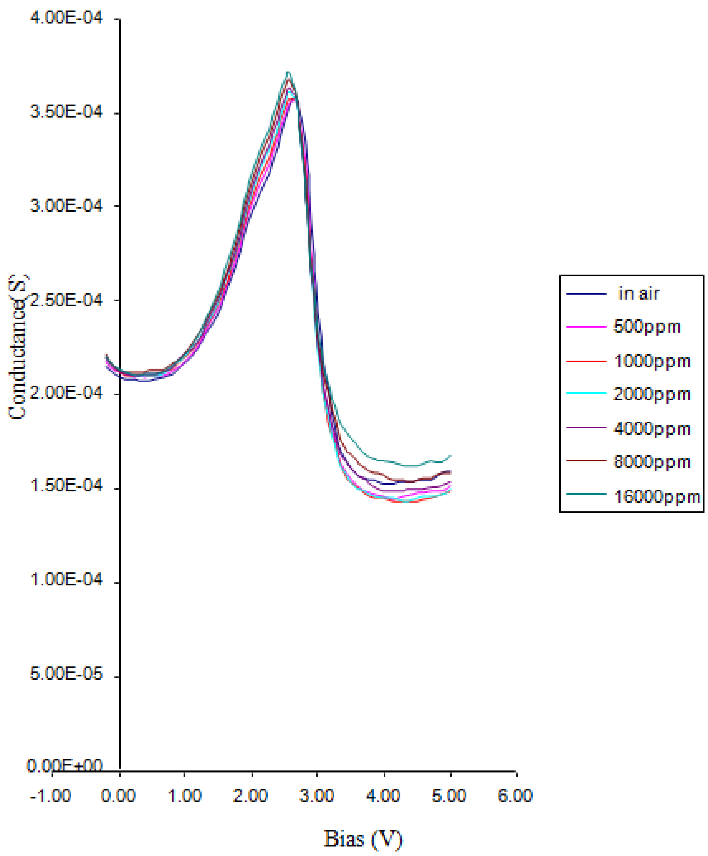

FIGURE 8: G-V Response of Pd gate MOS sensor at 120^0 C

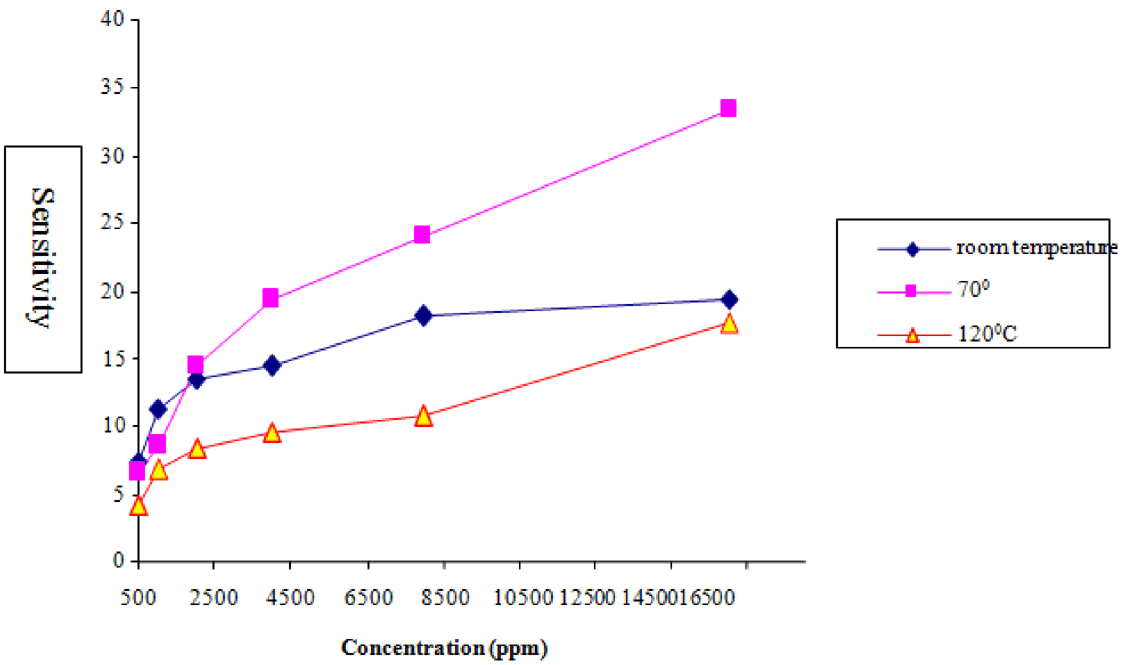

FIGURE 9: Variation of sensitivity with concentration at different operating temp. on exposure to Xylene

The variation of sensitivity with concentration at different operating temperatures is shown in fig. 9. The maximum sensitivity (33. 3%) is found at operating temperature 70˚C, where as for room temperature measurements the sensitivity reduces to 21. 6% at bias voltage of 0. 9 volts. The figures also reveal that flat band voltage of the sensor decreases by 200 mV at room temperature where as this change in V_{fb} becomes 380 mV at an operating temperature of 70˚C, which again shows that sensitivity of the sensor is much improved at this operating temperature.

3.2 G-V measurements

Figures (6-8) show the variation of conductance with bias voltage for the fabricated sensor, with varying concentration of xyelene, at different operating temperatures. These figures show that as the concentration of xyelene increases the peak magnitude of conductance increases and becomes saturated at particular concentration (1. 6 %). The conductance peak position also shifts towards left with Xyelene concentration. This shift is maximum (12. 5%) at an operating temperature 70˚C and reduces to 8. 42% at room temperature. Hence sensitivity of the sensor is again found to be highest at 70˚C if it is defined as

$$S(\%) = \frac{\Delta G}{G} \times 100$$

Where

G- Initial conductance peak position in air

∆ G- shift in conductance peak position at certain xyelene concentration

IV. DISCUSSION

From the previous works [1-4] it is well known that MOS capacitor with Pd gate show a lateral shift in the flat band voltage of the device on exposure to hydrogen gas. The hydrogen molecules are adsorbed and dissociated at the Pd surface. Some of the adsorbed atoms move at the Pd/ insulator interface, which give rise to change in electronic work function of the Pd surface due to their dipole moments. And therefore affects the flat band voltage of the device. When hydrogen gas is removed, the flat band voltage reduces to its original value [3].

In present work, the sensitivity of the sensor towards Xyelene vapors is probably due to catalytic dissociation of Xyelene molecules into hydrogen and carbon atoms. It can be believed that only hydrogen atoms have the accessibility to diffuse through the Pd layer to Pd/ SiO_2 interface, where they give rise to formation of dipole layer and change in flat band voltage of the device [3]. Dissociation of hydrocarbons on metals like Pd has been already reported by various researchers [8, 9]. The variational behavior of capacitance and conductance with temperature may be due to thermal generation of charge carriers, which increases with increase in temperature. Also the significant response of minority carriers at elevated temperature and contribution of induced surface traps to generation-recombination at higher temperature may be responsible for variation in C-V and G-V response of fabricated sensor.

At higher temperature, hot Pd surface in air, will act as dehydrogenation catalyst for Xyelene and will accelerate the dissociation process, resulting in greater number of available hydrogen atoms (as compared to room temperature) which are adsorbed at the metal surface to diffuse into the oxide layer and give better response. Hence sensitivity should increase with temperature as expected. However in present study, sensitivity is becoming maximum at 70˚ C and decreasing beyond this temperature. It can be accounted for by high concentration of surface states in the oxide layer and at "insulator/semiconductor" interface. Probably, the negatively charged oxygen ions which are adsorbed at the metal surface combine with H^+ to for OH^-which diffuses into the oxide layer to form an extra charge layer and ultimately modulating the surface states. At higher temperature, this process should increase but it seems that at 70˚ C the coverage of OH^- is saturated at the metal surface and beyond this temperature surface states modulation do not have a dominant effect on the sensitivity and now only the minority and thermally generated charge carriers have predominant contribution which results in fall in the sensitivity beyond 70˚ C.

The measurements also show that peak conductance value increases with Xyelene concentrations which can be attributed to the increase in interface charge density as reported by the numerous researchers (10-12) on exposure of hydrogen. In the present case also xyelene ultimately dissociates into hydrogen which increases the interface charges. The increase in peak conductance value is higher (8. 64%) at operating temperature of 70˚C as compared to the room temperature (3. 34%) can again be explained by considering the improved dehydrogenation at higher temperature and better availability of the hydrogen atoms at Pd/ SiO_2 interface.

V. CONCLUSION

It is concluded that Pd-gate MOS capacitor is a promising sensor for xyelene sensing. The highest sensitivity (33. 3% in terms of capacitance measurement and 8. 42% in terms of conductance) is found when operating temperature is 70^{o}C. The change in the flat band voltage of the sensor is also found to be greater (380 mV) at 70˚C as compared to operation at room temperature (200mV) when Xyelene concentration is varied from 500 ppm to 16,000 ppm.

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