Novel sensing approach for LPG leakage detection: Part I: Operating Mechanism and Preliminary Results

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Abstract—Gas sensing technology has been among the topical research work for quite some time. This paper showcases the research done on the detection mechanism of leakage of domestic cooking gas at ambient conditions. MEMS-based interdigital sensors were fabricated on oxidized single crystal silicon surfaces by maskless photolithography technique. The electrochemical impedance analysis of these sensors was done to detect liquefied petroleum gas (LPG) with and without coated particles of tin oxide (SnO_2) in form of thin layer. A thin-film of SnO_2 was spin-coated on the sensing surface of the interdigital sensor to induce selectivity to LPG that consists of a 60/40 mixture of propane and butane respectively. The paper reports a novel strategy for gas detection under ambient temperature and humidity conditions. The response time of the coated sensor was encouraging and own a promising potential to the development of a complete efficient gas sensing system.

Keywords—*Interdigital sensors, LPG, electrochemical impedance spectroscopy, coating.*

I. INTRODUCTION

Liquefied Petroleum Gas (LPG) had been used for last six decades, initially as an industrial fuel [1] and then for domestic cooking [2] and heating purposes [3] due to its smokeless burning in air. The main constituent of this gas is propane and butane mixed in a ratio of 60:40 normally [4]. However, the proportion of the two main gases may differ depending on specified application as well as seasonal condition. Gas leakage detection in residential houses has become one of the fundamental issues in recent times. Electronic and press media have reported some accidents caused due to gas leakage in residential houses [5, 6] and in industrial sectors [7, 8]. Usually, the accidents happen due to negligence rather than technical fault that cause gas to escape into the air.

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A better sensing system needs to be developed with enhanced limit of low concentration of gas to reduce accidents caused due to gas leakage. The gas is stored in pressurized metallic cylindrical bottles because its boiling point is lower than the ambient temperature. The gas is highly inflammable and is molecularly heavier than any other gas presents the air. Therefore, it settles closest to the ground level and keeps accumulating near the floor in the closed premises unless there is powerful exhaust system operating in the vicinity for its forceful disposal in the open atmosphere. It cannot be detected by human sense of smell due to its odourless nature. A pungent odour chemical is purposely added into the gas to make the presence of this gas detectable by human natural odour sensing system that facilitates human beings to sense its presence without any technical gadget. There are mainly two major drawbacks of such system. Firstly, it requires a human subject present in the vicinity. Secondly, by the time its presence in air could be sensed manually, its concentration exceeds a threshold level that is enough to cause an explosion by any spark triggering device like a light switch or metallic contact friction. In order to monitor its presence, sensors and sensing systems are deployed in LPG installed premises and environments to detect the leakage of gas and to avoid the accidents. There are some commercial gas detecting sensors [9] and systems available in the market that can issue warnings for a gas leakage incident. These sensors work on the concept of fixed controllers, fixed transmitters and fixed accessories. However, the cost of these commercialized sensing systems is high and has moderate technical complexity and inaccuracy with large delay in the response time. Therefore, a need for the development of a low-complexity, fast response and low-cost sensing system still exists.

A significant amount of research work has been executed previously by employing different types of sensors for gas detection [10-14]. But, it still is a hot topic of research due to the selectivity and sensitivity issues in the sensing mechanism and above-all long response and recovery time associated with the sensor. The work describes invoking a new methodology to improve the mentioned attributes.

The paper is divided into five sub-sections. After introduction in Section I, the working principle of the interdigital sensor

used for gas sensing and the experimental set-up are explained in section II. Results in section III show the response of the sensor towards the gas. The discussion part is given in section IV which explains the mechanism behind gas detection. The conclusion and the future work are given in section V inferring the work done and further experiments that need to be done to validate of the proposed approach.

II. INTERDIGITAL SENSOR AND EXPERIMENTAL SET-UP

2.1 Interdigital sensor: Working principle and fabrication

Interdigital sensors are used for the conduction of experiments. These sensors are based on the operating principle of parallel plate capacitors. The schematic diagram of interdigital sensor is shown in figure 1. The planar structure of the capacitor is formed for one-directional, non-invasive measurements. The planar geometry of the sensor introduces bulging electric field between the two plates in contrary to the generation of the uniformly parallel electric field in a co-axial form. When a time-dependent voltage signal is applied to the excitation, an electric field is generated between the two polarized plates as a result of the potential difference between them. Figure 1 clearly depicts the conversion of the parallel plate capacitor to planar form. The sensor can be used to measure the properties of material under test in many different ways. When any material is kept within the penetration depth of the electric lines of force of the sensing surface, the electric field passes through that material and gets modified by the material under test (MUT). The resultant electric fields which can provide information of the MUT are obtained from the measurement of sensor impedance.

Fig. 1: Schematic diagram of interdigital sensor [15].

The sensor chosen for the experiment was of 1-5-50 configuration. The reason for choosing such a configuration was to have a better resultant electric field from the excitation electrode to the sensing electrode [16]. This sensor has a configuration of 1-5-50, implying the presence of five sensing electrodes with respect to one excitation electrode, having a distance of 50 microns between two consecutive electrodes.

For this research, new sensors have been fabricated using maskless photolithography. Single crystal p-type silicon wafers with a thickness of 525 microns were selected for

constructing of interdigital sensors. An insulating layer of silicon dioxide was formed by thermal oxidation on these silicon wafers. The thickness of the oxidation layer was 500 nanometers. The wafer was coated with lift-off resistance (LOR) and positive photoresist, ECI 3027 followed by pre-baking the wafer at 180°C at 7 minutes and 100°C for 5 minutes respectively. The designs of the structure of the sensors were done using Windows supported L-Edit software, which was fed into a photolithography device for exposure of the patterns on the wafers. The wafer was then chemically, and plasma etched, followed by sputtering of metals of specified thickness on the etched patterns. The wafer was then sonicated, dried with nitrogen gas and finally cut into individual sensors. The fabricated sensor used for the experiment is shown in figure 2.

Fig. 2: Interdigital sensor having a configuration of 1-5-50.

To measure the capacitance of the sensor and consequently the dielectric permittivity, help of an electric circuit is taken. Figure 3 shows the equivalent circuit of the sensor along with a series resistance in presence of an electrical signal. The value of the series resistance will be very low so that it does not affect anything on measurement.

Fig. 3: Equivalent circuit of interdigital sensor.

The operating principle of the circuit for an interdigital sensor can be explained as follows:

$$V_{sense} = I_{in} * R_{series} \quad -- (1)$$

Where V_{sense} is the voltage measured across the series resistance and I_{in} is the current following through the sensor.

$$Z_{eq} = \frac{V_{in}}{I_{in}} = \frac{V_{in}}{V_{sense}} * R_{series} \quad -- (2)$$

Where Z_{eq} is the equivalent impedance of the circuit and V_{in} is the input voltage.

$$X_{sense} = Z * \sin \phi \quad -- (3)$$

$$R_{sense} = Z * \cos \phi - R_{series} \quad -- (4)$$

Where ϕ is the phase angle between V_{in} and I_{in} .

$$X_{sense} = w * C_{sense} \quad -- (5)$$

$$\text{And, } C_{sense} = \frac{\epsilon_0 \epsilon_r A}{d} \quad -- (6)$$

Where

C_{sense} = capacitance of the sensor in Farads, F.

ϵ_0 = the permittivity of free space ($\epsilon_0 = 8.854 \times 10^{-12} \text{ F/m}$)

ϵ_r = the relative permittivity or dielectric constant of the material under test (vacuum = 1).

A = effective area, square meters.

d = effective spacing between positive and negative electrode, meters.

Usually the phase angle ϕ between V_{in} and I_{in} is close to 90° for any dielectric material. The resistive part R_{sense} is usually very small or the change is insignificant. In normal dielectric characterization at low operating frequency, the X_{sense} is studied.

Electrochemical impedance spectroscopy (EIS) technique was used to determine the response of the interdigital sensors towards different concentrations of LPG. The electrochemical impedance spectroscopy technique owns high sensitivity with the limitation of stable and reversible system in equilibrium. Therefore, for non-stationary systems, instantaneous impedance measurements are mandatory. Electrochemical impedance spectroscopy and interdigital sensors had been used by a number of researchers like phthalate detection [17-20] in food, evaluation of nitrate contamination in water [21-25], for environmental monitoring [26], for meat fat content inspection [27, 28], moisture content estimation [29] etc. In the current research, the objective of the initial experiment is investigating response of the uncoated sensor to LPG at standard atmospheric condition.

2.2 Experimental set-up

All the experiments were conducted under identical temperature, humidity, and pH boundary condition in controlled laboratory environment. The sensor was connected to Hioki 3522-50 LCR Hi precision tester (Japan) by gold contact clamp pin connector. The spin coating technique described in the subsequent section was performed with a spin coater of 6800 series. The chemicals used for spin coating were brought from Sigma-Aldrich. Tin oxide (IV) had purity of 97% with a density 6.95 g/mL at 25°C (lit.). Ethyl alcohol had a purity of >99.9% with a flash point at 14°C .

The LCR meter was initially run with built in open and short calibration tests to nullify any stray capacitance due to connecting leads. LCR tester was set to slow mode of testing to achieve an error rate of <0.05% as recommended by the manufacturer. The sensor setup was formed in a desiccator to achieve the required controls of 44% humidity and inert atmosphere. To obtain a reference, the sensor was profiled in the air for a frequency sweep of 42 Hz to 2 MHz with ten measurements per decade change in frequency at log scale. No significant change of humidity was noticed during the experiments.

The detection of LPG was carried out inside a fume hood in a 5000 cm^3 airtight chamber with an inlet and an outlet. The interdigital sensor, placed inside the chamber, was connected to the LCR meter through a jig with gold-coated pressure contacts. The LCR meter was connected to the laptop through the serial communication port using RS232 interface. Window based data acquisition software was used to write the response signal in a Microsoft Excel file in the computer. The experimental set up is shown in figure 4. The gas used for the experiment was drawn from a 9 kg pressurized cylindrical bottle. Gas taken from this bottle was mixed with air from two separate tubes to attain a concentration of 20,000 ppm, which is the threshold for the explosion. The pressure of the gas and air were controlled at a flow rate of 700 ml/min and 20 lit/min by the two rotameters connected in order to achieve the desired concentration of LPG in the air.

The profile for the sensor in air was obtained for the range of real part ($R (\Omega)$) and the imaginary part of the impedance ($X (\Omega)$) to determine the nature of the sensor. The sensor was tested for a range of frequencies from 1 Hz to 300 Hz to obtain the optimal frequency of operation for the gas leakage detection. The test frequency for the experiments had been set to 15 Hz.

The characterization of the sensor was done in air and with different concentrations of gas to determine its response. Nyquist plot, i.e., the imaginary part of the impedance versus the real part of the impedance of the sensor for a range of frequencies has been shown in figure 5. It can be seen from the figure that the response of the sensor is quite indifferent to air and LPG. The sensing behavior of conventional interdigital sensor is not observed at all. There is a need to modify the surface of the sensor with some coating to induce sensitivity and selectivity.

Fig. 4: Experimental set-up for detection of LPG.

Fig. 5: Response of the sensor without coating towards air and gas.

2.3 Surface coating: Mechanism and principle

To induce the sensitivity as well as selectivity of the sensing surface, thin-film coating was done. There are different coating mechanisms available to achieve an LPG selective coating on the sensing surface of the sensor element, such as spin coating [30], dip coating [31] and electrospinning [32]. Spin coating mechanism was chosen among three mentioned coating techniques with the consideration of the following disadvantages of dip coating mechanism and electrospinning. The schematic diagram for spin coating is shown in figure 6. Firstly, unlike dip coating, spin coating technique forms a uniform layer of coated material. Secondly, the control over the coating thickness could be achieved more easily in comparison to electrospinning methodology. The sensor whose sensing surface was to be coated with a specific coating

material was placed on the circular metallic platform. Tin oxide (SnO_2) and ethyl alcohol has been used as the dispersed and dispersion medium respectively. It was then rotated at fixed revolutions per minute (rpm) and duration to generate a uniform layer over the sensing surface.

The time of spin coating plays a vital part in achieving the target coating thickness. The influence of spinning time was also investigated in this research that is described in detail in later section of the manuscript. The angular speed of the platform that was measured in rpm could be varied to control the coating thickness of the coating. Another factor that affects the coating thickness is the specific gravity of the suspension. These two factors will be investigated and presented in future research work.

Fig. 6: Schematic diagram for spin-coating process.

Numerous oxides have been studied to optimize the conductivity effect. Among them, SnO_2 has been regarded as a material with favorable unique properties like chemical and thermal stability, natural non-stoichiometry and a distinct property of better oxygen absorbance. The use of SnO_2 has been done in many gas detection [13, 33, 34] works due to its semiconductive properties. SnO_2 was used as a thin-film layer on the sensing surface of the interdigital sensors for the selective LPG detection. The oxidation of the reactive gases at semiconductor metal oxide (SMO) thin film surface induces changes in the electrical conductivity of the film. The absorbed oxygen produce oxide on the surface and formation of oxide in the form of a thin film on the coating surface induce sensitivity, as sensing is essentially a surface phenomenon. Here, an n-type semiconductor is used as a functional layer on the sensing area to induce redox reactions with the gas. The incident molecules of propane and butane get absorbed in the semiconductor coating layer liberated electrons as a result of chemisorption in the thin layer. Hence, a change is observed in the flow of current through the sensor that reflects a change of impedance of the sensor for the material under test.

The suspension was formed with SnO_2 using ethyl alcohol as the dispersion medium. A semi-solid porous suspension was formed on mixing the two mentioned compounds. SEM image of the coated sensing surface is shown in figure 7. The image was obtained from a FEI Quanta 200 model with an

accelerating voltage of 20 kV. The thickness of the coated layer was 2.155 microns. The value was an average of the measurement between the ones taken vertically from the center and the side of the sensing surface by an electron beam directed vertically from the top of the sensor.

Fig. 7: SEM image of the coated surface.

III. EXPERIMENTAL RESULTS

The sensor was tested for different concentrations of gas to determine its response. The gas concentration was varied at a range from 500 ppm to 20,000 ppm. Nyquist plot of the sensor for the different concentrations of gas are plotted to analyze the change in impedance with respect to each concentration. The response is shown in figure 8. It can be seen that there is a change between the impedance values and gas concentration. The highest impedance is corresponding to the lowest concentration inferring to lowest conductivity. As the concentration increases, the impedance changes while principally the resistance keeps on decreasing. It is seen that the change in real part of impedance is more than the imaginary part suggesting that the gas detection is taking place due to the change in conductivity.

The change in impedance, phase angle and real part of the impedance are shown in figures 9, 10 and 11 respectively. The readings are taken at low frequencies from 1 Hz- 300 Hz with 20 data points per decade on a logarithmic scale. The reason for considering lower frequencies is due to the charge transfer process that takes place which is explained in the subsequent section. The change in the impedance (Z) is more prominent in lower frequencies compared to higher frequencies. The change in the phase angle (Φ) is very low at lower frequencies up to an extent (around 10 Hz) and then keeps increasing exponentially with increasing frequencies. The real part of the impedance also shows subsequent change in the lower frequencies shown in figure 11. The impedance values changes symmetrically along with the concentration of gas. It can be seen that the resistance values changes almost symmetrically with respect to each concentration of gas. This

signifies the change in conductivity occurring at the coated surface of the sensor.

Fig. 8: Response of the sensor with coating towards different concentrations of gas.

Fig. 9: Change of the impedance (Z) with respect to frequency (1 Hz- 300 Hz).

Fig. 10: Change of phase angle (Φ) with respect to frequency (1 Hz- 300 Hz).

The sensitivity of the real part of impedance towards the gas is shown in figure 12. The change in % of sensitivity is calculated from the following formula:

$$\text{Sensitivity (\%)} = \frac{(R_{\text{control}} - R_{\text{measured}})}{R_{\text{control}}} \times 100 \quad -- (7)$$

It can be seen from figure 12 that the response of resistive values changes almost linearly with the concentration. This signifies that resistive part has a major role to play in the working mechanism of the gas detection.

Fig. 11: Change of real part of impedance (R) with respect to frequency (1 Hz- 300 Hz).

Fig. 12: % change of sensitivity of R with respect to different concentrations of gas.

IV. DISCUSSION

It can be inferred from the above results that the detection of LPG at different concentrations is taking place predominantly due to the change in resistance. A reaction between the coated sensor and the gas has taken place. The kinetics of charge transfer process at lower frequencies is more prominent than the ion diffusion process [35]. This can be explained from the structural change that occurs on reception of the gas. The structure of SnO_2 at room temperature is given in figure 13[36]. It can be seen from the lattice structure that the compound is chemically stable in the absence of traces of LPG. LPG undergoes chemisorption when exposed to the interdigital sensors coated with the semiconducting SnO_2 thin layer. The resistance of tin oxide thin films changes nonlinearly with the change in the surface temperature, thin film thickness, gas concentration and stoichiometry. A potential barrier mechanism dictates the magnitude of the electrical resistance offered by tin oxide coating in the thin film tin oxide based sensors. The bulk SnO_2 under the surface layer behaves as a 'molecular sieve' that prevents the oxygen adsorbed at the surface to diffuse into the bulk and holds it at the grain boundaries that leads to an electron depleted zone at the surface. The hydrocarbon gases such as LPG react with the adsorbed oxygen; consequently increasing the electron density in the SnO_2 surface that in turn increases the conductivity of the coating surface. The LPG acts as an oxidizing agent supplying surface oxygen ions to the conduction band of the thin-film coated SnO_2 layer, thus increasing the conductivity.

Fig. 13: SnO_2 structure at room temperature (normal condition).

Reduction reaction takes place for the long chains of propane and butane contained in LPG. A p-type response was conducted by the SnO_2 coating on LPG by attaching to the electrons from the gas resulting in an increase in current flow. The chemisorbed oxygen reacts with the chains of propane and butane leading to the release of electrons. These electrons released by the hydrocarbon chains are attached to the oxygen ions at the surface leading to the formation of a structure

shown in figure 14. This structure is chemically unstable compared to the normal SnO₂ structure. The inclusion of the electrons in the coated suspension layer signifies the decreases in impedance of the sensor in the presence of LPG. So the real part of the impedance is changing more compared to the imaginary part. But as the input voltage V_{in} is constant and R_{sense} is increasing, this will have an effect on X_{sense} and it won't be constant. But this change will be very nominal compared to R_{sense} .

Fig. 14: SnO₂ structure formation with LPG.

V. CONCLUSION AND FUTURE WORK

The paper presented a new approach in the detection of LPG at ambient conditions. Temperature and humidity conditions were maintained fixed during experimentation. The fabricated interdigital sensor was used to test different concentrations (in ppm) of LPG under coated and uncoated conditions to analyze the difference in response in two situations. The fabricated interdigital sensor was spin-coated with a thin-film of SnO₂ at fixed speed (1300 rpm) and timing (15 seconds) to induce a thin film layer of the sensing surface. The real part of the impedance changes more than the imaginary part as a result of exchange of electrons on the surface oxygen ions of SnO₂ on the sensing surface. The sensitivity of the real part of the impedance was calculated to determine the characteristic change in the response of the sensor with the variation of LPG concentration. The sensor showed a substantial change in the conductivity as a result of the exchange of electrons taking place between the gas and coated layer.

The next step would be to test the coated sensor on gas by varying the attributes of the thin-film surface like the concentration of the suspension layer along with the spin coating time and size of the dispersed medium of the

suspension. A low-cost, efficient gas sensing system would be developed to be considered for practical applications.

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