Development of Highly Sensitive Iron (III) Oxide Thin Film for Acetone Sensing

Mohd Nahid
Smart Sensors Area
CSIR-CEERI, Pilani,India
mohdnahid81@gmail.com

Vikas Saini
Smart Sensors Area
CSIR-CEERI, Pilani,India
vikasmawandia89@gmail.com

Jitendra Bhargava
Smart Sensors Area
CSIR-CEERI, Pilani,India
jbj@ceeri.res.in

Ashok Kr. Sharma
Smart Sensors Area
CSIR-CEERI, Pilani,India
aksharma@ceeri.res.in

Ajay Agarwal
Smart Sensors Area
CSIR-CEERI, Pilani,India
ajay@ceeri.res.in

Vinay Goyal
Smart Sensors Area
CSIR-CEERI, Pilani,India
vgoyal344@gmail.com

Rahul Prajesh
Smart Sensors Area
CSIR-CEERI, Pilani,India
rahulprajesh@gmail.com

Abstract—**Human breath contains various biomarkers which possibly can give us information about various diseases. Acetone is one such biomarker giving the information of blood glucose levels in the body. Breath acetone content for a healthy person is in the range of 0.5 to 2 ppm. Acetone concentration above this concentration may be an indication of person being diabetic. Hence it is imperative to detect this gas at low concentrations. This paper presents the development of iron oxide thin film for the highly sensitive detection of Acetone (CH_3COCH_3). Developed iron oxide thin film is characterized using FESEM and micro-Raman. It was demonstrated that the film exhibited a response of 65% for the acetone concentration as low as 1 ppm. The limit of detection for the film is ~50ppb.**

Keywords— RF sputtering, Raman, FESEM, Acetone, Highly sensitive, Iron Oxide

I. Introduction

Acetone also known as propanone or dimethyl ketone. Pure acetone is colorless and flammable liquid or gas. Acetone is a solvent and can be used for manufacturing plastic and other industrial products such as nail polish. Acetone is also present in cosmetics and many other household products. Textile industries uses acetone as a degreasing agent for wool and silk. Human body is a natural source of acetone, human body produce acetone as a by-product of metabolism. Human body uses sugar and glucose for energy, after eating food, hormone insulin moves out the glucose form bloodstream into the cell. When body does not make enough insulin or takes very few carbs than it uses glucose for fuel and burns fat. When lever breaks down fat, it generates a chemical in the body this chemical is known as acetone. Normal levels of acetone in breath lies in the range of 0.5 to 2 ppm [1], above this concentration a person may be diabetic with any one of its types, Type-I diabetes, Type-II diabetes and Type -III diabetes. The symptoms of acetone poising are low blood pressure, nausea, pain in belly, person may have a fruity odour, sweet taste in mouth, acting as if drunk, coma, drowsiness, stupor, difficulty in breathing, slowed breathing rate and increased need to urinate.

Thus, detection of acetone is very important, there are various types of gas sensing techniques available, one of them is chemi-resistive metal oxide gas sensor. Semiconductor metal oxide gas sensors are very popular because of their low cost and high sensitivity. Semiconductor metal oxide gas sensor becomes more attractive when the integrated as MEMS based metal oxide gas sensors. MEMS based gas sensors reduces power consumption as they have microheater fabricated on a single chip with interdigitated electrodes, these sensors are very compact in size and easy to mount with electronic circuit for controlling and automation [2].

Metal oxide such as Bi_2O_3, CeO_2, Co_3O_4, CuO, Fe_2O_3, Ga_2O_3, MoO_3, Nb_2O_5, NiO, SnO_2, ZnO, WO_3 etc are commonly used for gas sensing [3]. Iron oxide is one such material which is highly sensitive towards acetone even at low concentrations. Iron oxide is a n-type semiconductor material. The particle size and bandgap of iron oxide is temperature dependent, it shows direct bandgap of 1.75ev and indirect bandgap of 1.67ev after annealing at 400°C [4]. iron oxide is transition metal oxide with different phases such as FeO, α-Fe_2O_3, β-Fe_2O, γ -Fe_2O_3 and Fe_3O_4. We have demonstrated high sensitivity using iron oxide in hematite phase.

II. Experimental Details

A p-type Silicon wafer with thickness and orientation of 300 μm and <100> respectively is used as a substrate. The substrate wafer is piranha cleaned followed thermal oxidation (1 μm) for electrical isolation of iron oxide material from silicon substrate. RF magnetron sputtering system with iron oxide target of 99.99% purity was used for the deposition of iron oxide thin film. Thin film is deposited at 150W RF power with 5 mtorr pressure for 15 minutes. The thickness of iron oxide thin film is measured by surface profiler and found to be ~50nm. This Iron oxide deposited thin film is annealed at 400°C temperature for 5 hours for defect annealing and material stabilization.

Various characterizations such as Surface Profiler, SEM, RAMAN are used for the analysis of the thin film, this is important because the sensing performance of the sensor is dependent on the deposited sensing material properties. Gas sensing characterization of the sample was carried out using computerized mass flow controller based (MFC) system. Sample was mounted on a hot chuck integrated with a closed chamber. The sensing is carried out at 300°C. Baseline of the sample was taken in zero air ambient, where the sample surface get the adsorption of various oxygen species (O_2^- O^- O^{2-}) making film's resistance increase. O_2^- is formed below 100°C, O^- is formed from 100°C to 300 °C and O^{2-} is formed above 300°C [5]. The gas sensing setup [6] used for the gas sensing is shown in the figure 1.

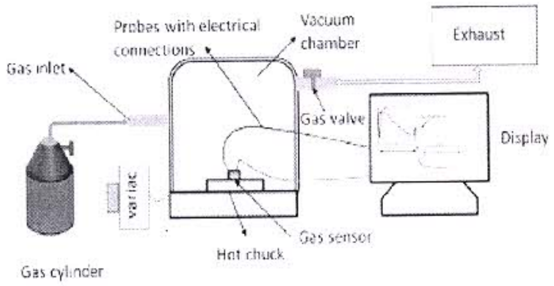

Figure 1 Schematic of the gas sensing setup used for characterization

III. RESULTS AND DISCUSSION

The FESEM (Field emission scanning electron microscopy) has been taken as shown in figure 2. It is clear from the SEM image that the deposited film is granular in nature which is preferred for gas sensing applications. As grain boundaries provides active sites for oxygen species to adsorb which makes it more sensitive toward the test gas.

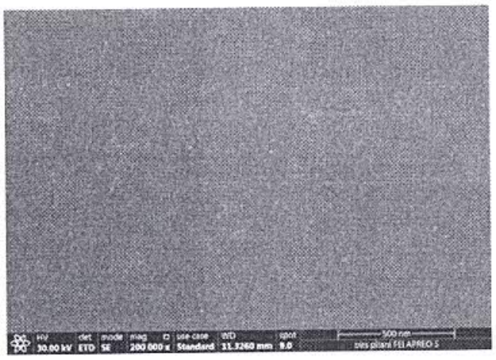

Figure 2 FESEM image of Iron oxide thin film

Raman characterization of the deposited film depicts that film contains bands related to hematite phase (α-Fe_2O_3) of iron oxide [7-11]. Intense peaks of the raman dictates that the sample has dominating hematite phase and bands related to other phases are hardly seen.

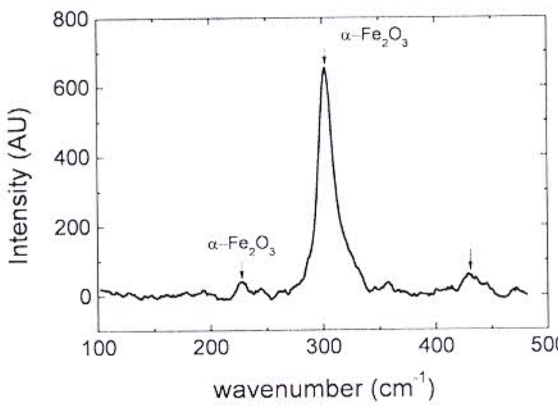

Figure 3 Raman spectra of Iron oxide thin film

Thin film is preheated at 300°C in the presence of synthetic air so that the oxygen molecules can adsorb onto the surface of sensor these oxygen molecules takes the electron from the conduction band of the sensor material and forms electronic species (O^- O_2^- and O^{2-}) as a result resistance of the sensor increases. On the exposure of acetone gas, it reacts with the adsorbed oxygen species on the sensor film and the electrons are released in the material with the formation of CO_2 and H_2O and resistance of the sensor starts decreasing. The response of the sensor is calculated with the following formula,

$$S = \frac{R_a - R_g}{R_a} \times 100 \ \% \qquad (1)$$

Here S is the sensor response, R_a is resistance in air and R_g is the resistance in gas.

Table 1Sensor characteristics for Iron Oxide thin film sensor

Acetone (ppm)	Temperature °C	Response (%)
1	300	65
3	300	73
5	300	78

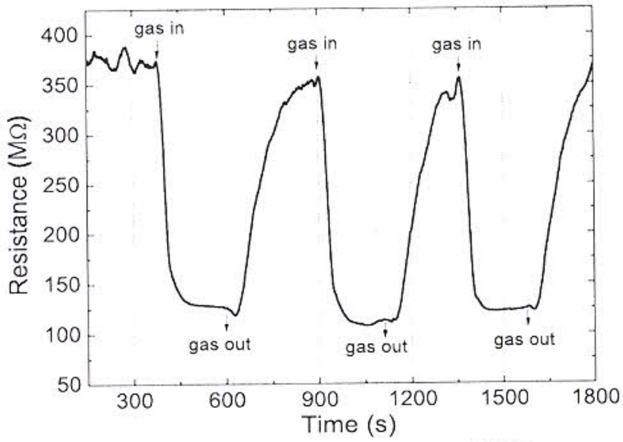

Figure 1 sensor characteristic curve for 1ppm Acetone at 300°C temperature

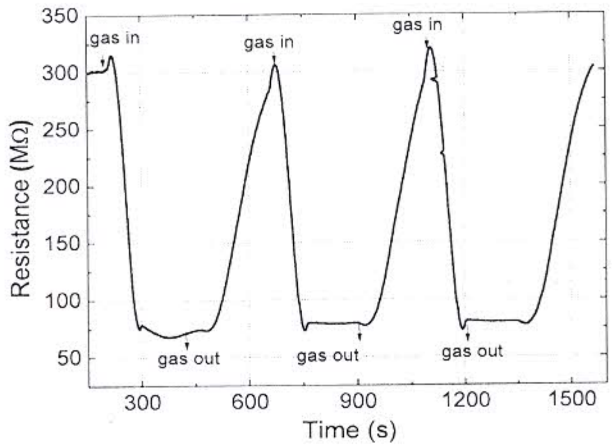

Figure 2 sensor characteristic curve for 3ppm Acetone at 300°C temperature

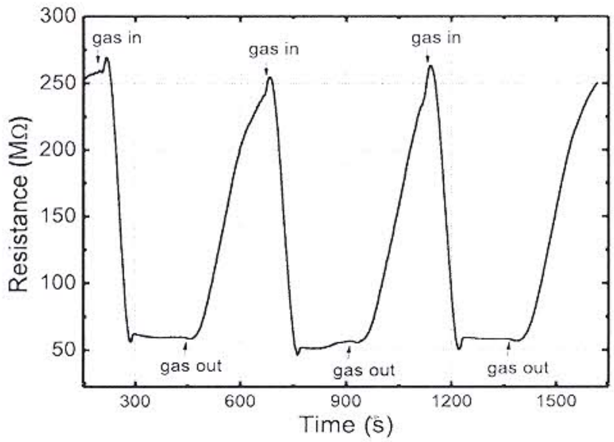

Figure 3 sensor characteristic curve for 5ppm Acetone at 300°C temperature

The sensor has been characterized at different temperatures from 200°C to 300°C, and this has been found that it is highly sensitive at 300°C temperature. Sensor also characterized at different concentrations of Acetone ranging from 1ppm to 5ppm. Response for all these concentrations is shown in table.1 and the sensor characteristic curve are shown in figure 4 – 6.

IV. Conclusion

The deposition of Iron(III) oxide thin films for Acetone sensing has been demonstrated successfully by RF magnetron sputtering system with deposited film thickness of ~50nm. To study the material thickness and surface morphology various characterization techniques such as Surface Profilometer, Raman spectroscopy, FESEM etc. have been performed. The sensitivity of Iron oxide based semiconductor metal oxide gas sensor is good, and response of the sensor is 65%, 73%, 78% for 1ppm, 3ppm, and 5ppm respectively. Developed film is highly sensitive towards acetone, hence it can potentially be used for non-invasive diabetes detection from breath.

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