A nanoporous thin-film miniature interdigitated capacitive impedance sensor for measuring humidity

T. Islam\textsuperscript{a}\textsuperscript{*}, Upendra Mittal\textsuperscript{a,b}, A.T. Nimal\textsuperscript{b} and M.U. Sharma\textsuperscript{b}

\textsuperscript{a}Department of Electrical Engineering, Faculty of Engineering & Technology, Jamia Millia Islamia (A Central University), Maulana Mohammed Ali Jauhar Marg, New Delhi 110025, India; \textsuperscript{b}Solid State Physics Laboratory, Lucknow Road, Timarpur, Delhi 110054, India

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This paper presents a development of a low-cost miniature humidity sensor with an interdigitated aluminium electrode connected in parallel on quartz substrate. Interdigitated capacitive device has been fabricated using the photolithography method. The aluminium electrode was covered with sensitive film of a nanoporous thin film of $\gamma$-Al$_2$O$_3$ made from novel sol–gel technique. Nanostructured thin film offers very high surface to volume ratio with distribution of micro pores for moisture detection. Pore morphologies of the film have been studied by field emission electron microscope and X-ray diffraction methods. Impedance measurement of the miniature capacitive humidity sensor toward relative humidity was investigated at room temperature by Agilent 4294A impedance analyzer (Agilent, Santa Clara, CA, USA). The device exhibits short response and recovery times and good repeatability.

Keywords: $\gamma$-Al$_2$O$_3$ film; interdigitated capacitive device; impedance; humidity sensor; sensing properties

1. Introduction

Humidity detection has always been an important task in the fields of automotive industry, food processing, meteorology, semiconductor technology, civil engineering, medicine and health care, industrial and agricultural production, environment protection, etc. Conventional techniques of humidity measurements are mechanical, chilled mirror hygrometers, wet and dry bulb psychrometers, infrared (IR) optical absorption hygrometers, capacitive and impedance type hygrometer. Various fiber optics-based techniques, that is direct spectroscopic, evanescent wave, in-fiber grating and interferometric methods, have also been used for humidity sensing. As technology improved the demand for low cost, reliable, quick response, and compact electronics circuit-based devices increased. Continuous efforts have been devoted to the research and development of humidity sensors to improve the sensing characteristics and miniaturize the size [1–4]. Recently, surface acoustic wave (SAW) humidity sensors became an attraction for researchers as they exhibit the advantages of high sensitivity, very small size, integrated electronic circuitry, easy-to-realize wireless communication, and so on [5–10]. SAW sensor with hygroscopic material coating makes excellent miniaturized humidity sensors having high sensitivity and fast response (few seconds), for real-time measurement of humidity over...
wide dynamic range. Mainly three working mechanisms contribute to the sensor output: mass loading, acoustoelectric, and viscoelastic effects [11]. Each mechanism can be effectively controlled for making an accurate low-cost humidity sensor. SAW sensor also has advantage as it can circumvent the problems of detecting very high impedance at dry atmosphere encountered for the impedance-type humidity sensors. It is known that the SAW humidity sensors usually have a high operating frequency in order to be smaller and more sensitive [8,11,12]. However, it creates a problem in the preparation of uniform humidity sensitive films with suitable thickness having very small sensing area. Otherwise, it may add to the cost of sensor preparation and affect the consistency in mass production. In last few years, a different kind of SAW impedance sensor had been developed and used in humidity measurements or other applications [13–15]. As we know that chemically, thermally, and mechanically γ-Al₂O₃ is a highly stable material, this nanoporous metal oxide will be suitable for humidity measurement. It is possible to make the nanostructure by chemical anodization, template and low-temperature sol–gel method. Pore morphology can be tuned to fabricate humidity sensor for different working range from few traces of moisture to percentage relative humidity [16,17]. In the recent past, several works have been reported by the authors and some other groups to utilize the material for both trace and RH level humidity measurement [18,19]. Some of the commercial dew point meters for industrial applications employ thin-film porous aluminium oxide-based capacitive technique [18].

In the present work, we have prepared an interdigitated capacitive impedance humidity sensor. An interdigitated capacitive device with sensitive film of γ-Al₂O₃ with 300 MHz delay line has been fabricated with standard lithography process. Each interdigitated Al electrode (IDT) for the capacitive device is deposited on the substrate. The sensing film is deposited on the electrode by dip coating of sol–gel solution of γ-Al₂O₃. Since the sensing film on IDT forms an interdigital capacitive humidity sensor, experiments have been conducted to observe the humidity response of the impedance-type sensor and it shows good response. Effects of the sensitive film on the sensitivity, repeatability, and response time of the sensor are also examined. Proposed interdigitated capacitive sensor is in miniaturized form and it can be batch fabricated with identical characteristics using existing integrated circuit fabrication technology. Also material requirement for sensing film is very small. Thus, it is possible to fabricate the device at low cost.

2. Sol solution and microstructures of the porous γ-Al₄O₃

Coating sol solution of γ-Al₂O₃ has been prepared by hydrolyzing the mixture solution of aluminium sec. butoxide (C₁₂H₂₇AlO₃) and water and subsequently peptizing the colloidal solution by adding concentrated hydrochloric (HCl) acid. The solution is then refluxed and the binder polyvinyl alcohol is added. Details of preparation of sol are reported elsewhere [20,21]. To determine the pore morphology of the γ-Al₂O₃ a thin film has been prepared on ST-X quartz substrate by dip coating method and film has been sintered at 400°C for 4 h. The morphology of the γ-Al₂O₃ nanostructure film was characterized by X-ray diffraction (XRD) and field emission electron microscope (FESEM). The main object of the XRD plot is to establish the formation of thin film of γ-Al₂O₃ on quartz substrate. Figure 1(a) shows XRD results for γ-Al₂O₃ film deposited on quartz substrate while Figure 1(b) shows XRD result for γ-Al₂O₃ film deposited on alumina substrate (alpha). Figure 1(c) shows XRD result of quartz substrate only without film. When we make a thin film of γ-Al₂O₃ on Quartz, the XRD peak is obtained at 2θ of 50°. However, the XRD peak of pure quartz material
(without γ-Al₂O₃ film) is obtained at 2θ of 28°. This study has confirmed the formation of γ-Al₂O₃ film on Quartz substrate.

Figure 2 shows the FESEM image of the porous structure of the sample at low magnification (scale = 100 nm), which suggests that the pores are almost spherical in shape with smooth surface. Pore size has been measured with the help of FESEM at very high resolution (432.390 K X). We have observed the distribution of pores size in the range of 5–10 nm as shown on the photograph. The measurement of pore size has been done using the software available with Carl Zeiss FESEM (model-Supra 55; Carl Zeiss SMT Inc., Peabody, MA, USA) instrument. The average pore size is found to be ~10 nm.

Figure 2. Field emission scanning electron micrograph of the nanoporous structure of γ-Al₂O₃ film deposited on quartz substrate.
2.1. Fabrication of the sensor

Performance of the capacitive impedance sensor depends on the (i) shape and dimension of the electrode, (ii) crack free $\gamma$-Al$_2$O$_3$ film on the device, (iii) thickness, and (iv) the uniformity of the film. An interdigitated electrode having 300 MHz SAW delay line device is fabricated using the photolithography process on ST-cut quartz substrate with zero temperature coefficient of delay. For the fabrication of the capacitive device, a circular quartz substrate of 3″ diameter and 500 µm thickness has been used. After proper cleaning of substrate, aluminium (99.999% pure) metallization has been done using the thermal evaporation technique. Using the wet etching process aluminium electrode IDE (inter digital electrode) has been obtained on the quartz substrate. Then the substrate has been diced and packaged. The size of IDE is 1.31 µm × 0.2 µm (electrode finger width × aluminium thickness) and the capacitive chip size is 5 mm × 8 mm × 0.5 mm. The gap between two adjacent fingers is 1.31 µm.

The device is packaged with a metal shell to ensure its stability. To deposit the sensing film, the device is properly cleaned in the argon plasma for removing the undesirable impurity. The $\gamma$-Al$_2$O$_3$ film is then deposited on the electrode as well as mass loading area by dipping the device with IDE two times in the sol solution. The device electrodes covered with the sensitive film are sintered in oven at 400°C for 4 h [18]. Thickness of the film has been optimized for the improvement in the sensitivity. The $\gamma$-Al$_2$O$_3$ film has been characterized using the SOPRA optical ellipsometry system (Sopra Inc., Palo Alto, CA, USA) for thickness measurement. The film thickness is found to be in the range of few hundreds nanometer (~200 nm). The photograph of the interdigitated capacitive sensor is shown in Figure 3.

3. Determination of sensor characteristics

3.1. Experimental set up

To obtain a rapid, accurate, and reliable response of the sensor to the different concentrations of humidity in the nitrogen gas, a computer-based data acquisition system is
employed. The sensor is put inside a rectangular shape steel test chamber of nearly 50 cc volume. The sensor chamber is fitted in series with a commercial Honeywell RH meter (Bombay Engineering, Kolkata, India). Humidity contained nitrogen gas is obtained by passing the dry nitrogen gas through water vapor. The amount of residual humidity in the dry N\textsubscript{2} gas is less than 4 ppm. The humidity level in the chamber is controlled by precision needle valve and it is measured by calibrated commercial RH meter. The accuracy of the commercial meter is \(\pm 2\%\) RH. The humidity level in the moist gas varied in the range of 20–97\% RH at room temperature of 25°C. The leads of the sensor were connected to an impedance analyzer (Agilent 4294A; Agilent, Santa Clara, CA, USA). The impedance analyzer was interfaced to a PC through a data acquisition system. The sensor was excited by AC signal of amplitude 500 mV (r.m.s.) and the frequency of 1 kHz. Here frequency of operation is used to design the finger width of the one IDE. In the present work, the IDE formed below sensing film is used as capacitive sensor. For capacitive moisture sensing, the low signal frequency of 1 kHz is desirable for high sensitivity [1,18]. Experiments were conducted at room temperature to examine the sensor parameters such as: (i) capacitive response, (ii) dissipation factor (\(D\)), (iii) impedance response (\(Z\)), (iv) transient response for response and recovery times, and (v) repeatability of sensor response output.

### 3.2. Electrical characteristics of the sensor

Capacitance change with the variation of humidity is shown in Figure 4. The capacitance value increases with increase in humidity. It has been observed that if the signal frequency is increased, capacitance change with humidity becomes smaller. Dissipation factor of the capacitor is shown in Figure 5. Impedance change of the sensor with change in % RH is shown in Figure 6. Since the film is made of metal oxide at low humidity it shows very high impedance and as humidity increases, it decreases rapidly. Response and recovery times are other important parameters of the sensor which are determined from the transient response curve. The response time is the time taken by the capacitive sensor to change the

![Figure 4](image-url)  
Figure 4. Capacitance change of the thin-film capacitive sensor with variation of humidity in the range of 20–97\% RH.
output from 10% to 90% of its maximum value while the recovery time is the time taken by the sensor to return from 90% output to 10% of its initial value. For real-time application, these parameters should be as small as possible.

Figure 7 shows the transient response curve for 87–20% RH changes in humidity. The response and recovery times are approximately 15 and 75 s, respectively. The repeatability of the sensor output for the same humidity change for several cycles is shown in Figure 8. Only capacitive type humidity sensor fabricated on quartz has been tested and their repeatability is very good and its value is close to 100%.

4. Results and discussion

Table 1 summarizes the experimental results for the miniaturized capacitive sensor. Figure 4 shows the capacitance change of the thin-film sensor with variation of humidity. Capacitance value increases monotonically in the beginning but there is a sharp change in the value above 50%. The capacitance change is almost linear in the range of 50–80% RH.
and above 80% RH, it increases slowly. When the sensor is exposed to humidity, the vapor molecules initially adsorb on the surface and then condensed in the nano-order pores causing effective change in dielectric constant. But since the sensing area is very small, the capacitance change at lower humidity is small and as humidity increases enough vapor molecules condensed in the porous layer causing large change in dielectric constant. Also at higher humidity above 80%, many of the pores are already or nearly filled up causing lesser change is dielectric constant [18]. Figure 6 shows the impedance

![Figure 7](image_url)

Figure 7. Transient response of the sensor to the humidity change from 20% to 87% RH.

![Figure 8](image_url)

Figure 8. Repeatability of the sensor output from 20% to 87% RH change in humidity.

| Table 1. Electrical characteristics of the sensor. |
|---------------------------------------------|
| Operating moisture range | 20–87%RH |
| Nominal capacitance | 100 ± 1 pF |
| Sensitivity | 7.2 pF/%RH |
| Response time (t90) | 15 s (20–87%RH) |
| Recovery time (t90) | 75 s (20–87%RH) |
| Nonlinearity | 1.05% (max [(C/Cmax) × 100]) |
| Temperature coefficient | 0.04 pF/°C (22–85°C) |
| Operating frequency range | 1–100 kHz (suitable frequency = 1 kHz) |
response of the sensor for increase in RH. The impedance change is due to change capacitive reactance as well as conductivity of the water adsorbed. Initially water molecules are chemically adsorbed then physically attached on the chemically adsorbed layer. The decrease in reactance is due to the increase in capacitance and the increase in conductivity is due to increase in protons’ (H\(^+\)) concentration of the sensor with vapor. The adsorbed OH\(^-\) ions provide easy path for proton hopping \[16\]. Also the small gap between the fingers plays important role for sharp change in impedance. Because of very thin sensing layer, the response is faster and hysteresis error is found to be negligible. Hysteresis is one of the concerns of any humidity sensor working on adsorption and desorption phenomenon. Hysteresis error arises due to incomplete adsorption and desorption of water molecules, and for a good sensor it should be as small as possible. The hysteresis error depends on the pore morphology and thickness of the sensing layer \[1,18\]. Since the sensor is based on thin film of metal oxide, the output drift is considerably smaller \[16\]. Transient response of the sensor is shown in Figure 7, and the sensor shows very fast response time (15 s). The response behavior is governed by the pore morphologies, and the morphologies are governed by the average dimension of the pore, the distribution of the pores, thickness of the porous layer, and stability of the sensing film \[1\]. Pore morphology plays a significant role in the condensation of water in the porous matrix. Since the mean free path and size of incident moisture cluster varies with the change in humidity concentration, an optimum pore size comparable to mean free path is desirable for humidity sensing. The response also depends on the signal frequency, which is related to orientation loss of humidity. The interdigital capacitance \(C_{IDT}\) shown in Figure 9 can be determined from the following equation \[22\].

\[
C_{IDT} = C_{UC}(N - L)
\]

where \(C_{IDT}\) is the capacitance of the IDT, \(C_{UC}\) is the capacitance of unit cell, \(N\) is the number of electrodes, and \(L\) is the length of the electrode. \(C_{UC}\) can be written as

![Figure 9. The interdigital electrode structure of the capacitive impedance sensor.](image)
The capacitance $C_1$ and $C_2$ are calculated by conformal mapping $K[x]$ of the two semi-planes with dielectric constants $\varepsilon_1$ and $\varepsilon_2$ into two parallel-plate capacitors.

$$C_1 + C_2 = \varepsilon_0 \frac{\varepsilon_1 + \varepsilon_2}{2} \left[ K \left( \frac{a^2}{b^2} \right) \right]^{1/2}$$

where $a$ is the gap between the electrodes, $b$ is the single electrode width. The capacitance $C_3$ is given by

$$C_3 = \varepsilon_0 \varepsilon_3 \frac{h}{a}$$

where, $\varepsilon_3$ is the effective dielectric constant of the sensing film in the presence of water, $h$ is the thickness of the electrode. As a (gap between the electrodes) is inversely related with $C_3$, decreasing the gap between the electrodes or increasing the number of fingers (actual sensing area) may increase sensitivity toward lower humidity range. Reliability of the sensor system is shown in Figure 8. As our capacitive type humidity sensor has been tested with same humidity several times and it gives the same output in every cycle as the $\gamma$-Al$_2$O$_3$ film coated capacitive device is very stable and repeatability of the sensor system is very good. It has been reported that $\gamma$-Al$_2$O$_3$ film is highly stable thermally, hence the effect of ambient temperature variation is negligible [18].

5. Conclusion
In the present work, a miniaturized interdigitated capacitive device using thin film of nanoporous alumina for humidity sensing has been fabricated. The humidity sensing film has been prepared by sol–gel technique. Since the sensing film on the IDE of the device forms an interdigital capacitive impedance sensor, initial experiments have been conducted to study the impedance response with 20–97% RH change in N$_2$ gas. The achieved characteristics are suitable for employing the sensor to measure humidity in the prescribed range. The response characteristics are significant, fast, and highly reproducible. However, the sensitivity can be increased in the lower humidity range by increasing the area of the interdigital electrode and sensing film of the device. The actual sensing area is only 5 mm × 8 mm.

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