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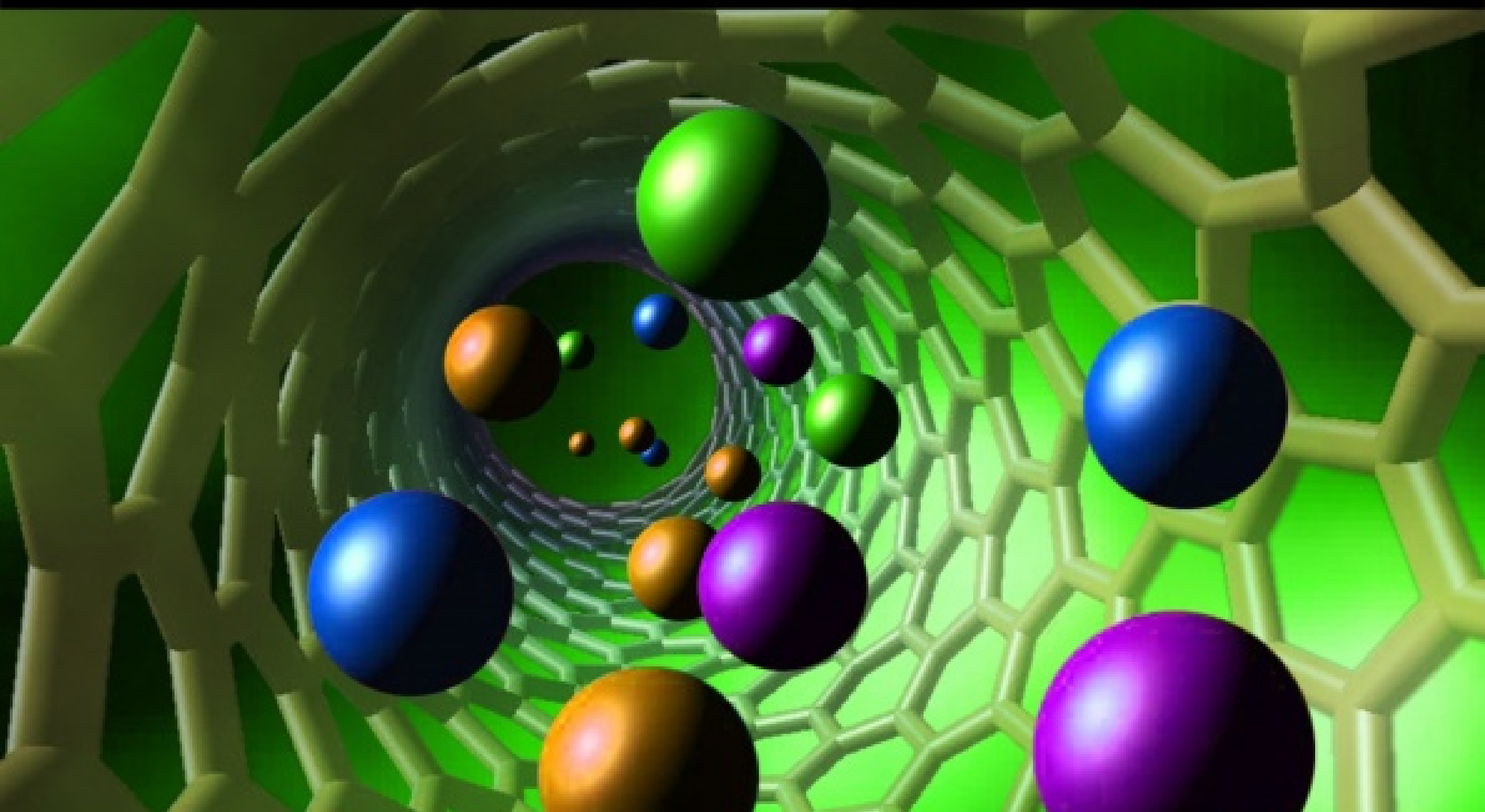

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Electrical Characterization of a Nanoporous Silicon Sensor for Low ppm Gas Moisture Sensing

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Abstract: A nanoporous silicon sensor prepared by electrochemical etching of p type single crystal silicon in HF electrolyte has been characterized for measuring gas moisture in the range of 6 to 100 ppmV. Impedance characteristics show that PS may also be useful for developing CMOS compatible trace moisture sensor. The behavior of the capacitive sensor has also been inverse modeled using multilayer perceptron neural network to determine the concentration of the soft sensor. The simulation results closely follow the actual sensor response. *Copyright © 2007 IFSA.*

Keywords: Porous silicon, ppmV moisture sensing, Electrical characterization, Inverse modeling, Multilayer perceptron neural network

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1. Introduction

Presence of moisture in undesirable quantities can have detrimental effects on many industrial processes such as petrochemical, natural gases, pharmaceutical and semiconductor industries. In the absence of an accurate and desirable sensor to measure moisture, it has become industry practice to rely on expensive workaround and over designed systems to eliminate moisture allowing huge safety margins. Thus there is an increasing demand of suitable moisture sensor to detect the trace level moisture concentration in the gases. Moisture sensors must fulfill a number of requirements: they have low respond time, high sensitivity, and accuracy, their temperature range should be as large as possible, free from aging, contaminants and they should preferably be selective with respect to other vapors and gases [1]. Various moisture sensors like electrolyte, organic polymer, ceramic and alumina thin films [2-3] have been commonly used to measure moisture content in gases at the ppm level. Although bulk sintered ceramic materials showed stable physical and chemical properties, they suffer from irreversible response and large recovery time due to chemisorptions and adsorption of water molecules [2-3]. Recently, porous alumina prepared by sol-gel technique has been reported to fabricate capacitive trace moisture sensor in the ppm level [4]. This sensor is sensitive to gas moisture in the range 5-200 ppmV. However, the ceramic-based sensor poses difficulties of IC compatibility and fabrication. The PS (porous silicon) sensor is an electrochemical derivative of silicon in HF based electrolyte, have many useful properties for sensor applications such as large surface to volume ratio, controllable pore morphology and pore structure for high sensitivity, selectivity and rapid response time [5]. By specifying particular etching parameters, pore dimensions such as micro pores (1-4 nm), meso pores (4-50 nm) can be finely controlled. Different pore sizes and pore morphology suite different molecules, so these can be tailored to the sensing capability required. More over, it has proven practical to produce pores of graded size on the same substrate to achieve multiple targets sensing capability on the same chip [5]. Based on graded morphology using suitable formation parameters, a porous silicon based vapor sensor array for sensing organic vapors was developed [6]. Another attraction of using porous silicon is that the PS sensor is CMOS IC compatible. Vapors or gases penetrating into its pores can affect several physical property of PS such as conductivity, dielectric constant, and photoluminescence. Utilizing these parameters different types of sensors like gas, vapour, and pressure have been developed [1, 6]. Several research articles have been are reported for fabricating relative humidity sensor but little work has been reported to detect traces of gas moisture in the lower ppm range using porous silicon. However, porous alumina deposited on SiO_2/Si substrate has been used for measuring humidity in the range of 0.1% to 1 % [7]. Very recently, an attempt was made to develop porous silicon gas moisture sensor in the range from 6 to 200 ppmV [8]. It was reported that PS could also be used to sense trace moisture in the gases though the sensitivity in comparison to porous alumina is not sufficient which, can be improved further by optimizing the pore morphology and pore structure [5].

We present in this paper the preliminary ac electrical characterization of a nanostructured porous silicon based ppm level gas moisture sensor in the range of 6 to 100 ppm. Impedance characterization is now widely used to understand the operation of sensors [7, 9]. The study of dielectric relaxation of absorbed moisture for capacitive moisture sensor is also important [10]. The sensing mechanism is analyzed in terms of dielectric loss properties for ppm level moisture sensor. To estimate the gas moisture concentration the characteristics of the PS sensor is function approximated using artificial neural network (ANN) [11-12]. Though the polynomial interpolation can also be used for the same purpose but ANN based approach which is adaptive for modeling the sensor behavior, which is sufficiently nonlinear is a suitable choice [13]. Behavior modeling of the sensor using ANN technique can help to determine the faulty operation of the sensor [14].

2. Fabrication of the Porous Silicon Sensor

For the characterization of the PS sensor for detecting the moisture concentration in ppm level, the porous silicon moisture sensor has been fabricated on silicon wafer (2.5cmx2.5cmx0.5mm), with <100> orientations having a resistivity of 1-2 Ωcm by standard electrochemical etching in hydrogen flouride (HF) based electrolyte. For nanocrystal PS, back metal contact has been established by screen-printing technique with Ag-Al paste and subsequently fired at 700 °C for 45 sec in inert gas atmosphere. The silicon wafer is then anodized in a teflon bath which is PC interfaced through data acquisition system for precisely controlling the current density and etching time. Fig. 1 shows the photograph of the PC interfaced PS formation cell.

Fig. 1. Photograph of the PC interfaced porous silicon formation cell.

The formation parameters used to obtain nanocrystal of PS for sensor were; HF concentration = 48 %, current density = 10 mA/cm^2, etching time = 1 m respectively. The porosity of the PS sample was determined gravimetrically by using the relation [9]:

$$P = \frac{m_1 - m_2}{m_1 - m_3}, \quad (1)$$

where, P is the porosity, m_1 is the mass of the sample before anodization, m_2 is the mass after anodization, and m_3 is the mass of the sample after complete dissolution of the porous layer in dilute NaOH solution. The lateral metal contacts have been established by vacuum evaporation of aluminium on the porous layer. The sensor structure is membrane type [6]. The nano order pores have the Gaussian distribution within the range of 3-10 nm. The thickness of the PS layer is of the order of 5-7 μm.

3. Sensor Testing

Experimental set up used for sensor testing is reported elsewhere [6]. The test chamber consists of a cylindrical steel case 10 cm in diameter and 12 cm high. Chamber was fitted with inlet and outlet gas pipelines. The inlet pipe was also connected to a standard thin film alumina moisture sensor to allow an equivalent amount of gas flow to the standard commercial sensor. PS sensor is placed inside the chamber, and the sensor is connected with shielded cable 2 ft long. PS sensor is put in a conducting shield (steel case) and the shield is grounded to keep the parasitic capacitance constant and thus sensor electrodes can be shielded from external electric fields. The chamber was purged with dry blended air at a flow rate of 4 l /m. The sensors were allowed to equilibrate for several hours prior to testing. All the electrical characterization of the sensor is taken using a Solatron Impedance analyzer. The output of the impedance analyzer is interfaced with a PC through data acquisition system. The excitation voltage for the sensor was fixed at 1 V (rms) and the signal frequency was varied from 100 to 100 kHz, respectively. Measurements were made at room temperature of 25 °C. Each measurement has been taken giving sensor at least 20 minutes to reach equilibrium state. The following procedures were adopted. (i) Initial concentration of moisture is adjusted to 6 ppmV using the standard moisture sensor, (ii) capacitance/impedance/phase value of each sensor was noted, (iii) test gas mixture was injected into the chamber and exposed to the sensors, (iv) electrical parameters values of the sensors were measured, (v) concentration of moisture in the gas was increased from 6 ppmV to 100 ppmV and the values of parameters were measured.

4. Experimental Results

Experiments are conducted to characterize the behavior of porous silicon sensor for the variation of gas moisture in the range of 6 to 100 ppmV. Measurements are taken to see the effect on frequency on the electrical parameters. In one experiment, we find the change in impedance both with the variation of gas moisture from 6 to 100 ppm and frequency from 100 Hz to 100 kHz. This impedance plot is shown on 3D graph in Fig. 2. The change in impedance is strictly decreasing both due to moisture concentration and signal frequency.

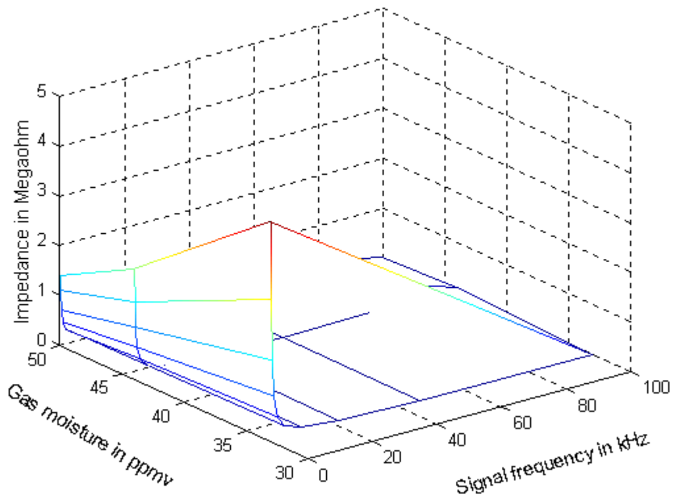


Fig. 2. Impedance change of PS gas moisture sensor for the variation of both moisture content and signal frequency from 100 Hz to 100 kHz (Solatron Impedance analyzer, 1 V rms).

Fig. 3 shows the dependence of phase on the moisture content and frequency. It is observed from Fig. 4 that as the moisture concentration increases from lower to higher, the phase shift also increases from its initial values. As the concentration is increasing effective dielectric constant as well as conductivity is increasing resulting in phase shift from its initial value. Fig. 4 shows 3D plot showing the variation of capacitance for the variation of moisture and signal frequency. It can be seen that sensor shows strong sensitivity. Due to increase in moisture concentration the effective dielectric constant is increasing resulting increase in capacitance change. However, with increasing in frequency the capacitance change decreases. This is due to the fact that dielectric constant is frequency dependent quantity and it decreases with frequency [15]. The capacitance of the material with conduction can be expressed as [10]

$$C = \varepsilon_{eff} C_0 - i\frac{\sigma}{w\varepsilon_0} C_0 , \qquad (2)$$

where ε_{eff} is complex dielectric parameter, C_0 is the capacitance of ideal capacitor, ε_r is the relative dielectric constant of the medium, ε_0 is the dielectric constant of vacuum , σ is the conductivity, w is the signal frequency. Thus the actual capacitance of the sensor in moisture medium is proportional to conductivity and inversely proportional to the signal frequency. It is obvious in Fig. 4 that as frequency increases capacitance decreases.

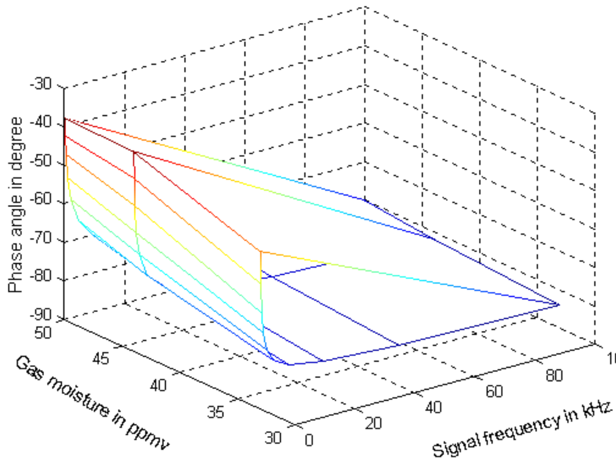


Fig. 3. Phase angle change of PS gas moisture sensor for the variation of both moisture content and signal frequency from 100 Hz to 100 kHz (Solatron Impedance analyzer, 1 V rms).

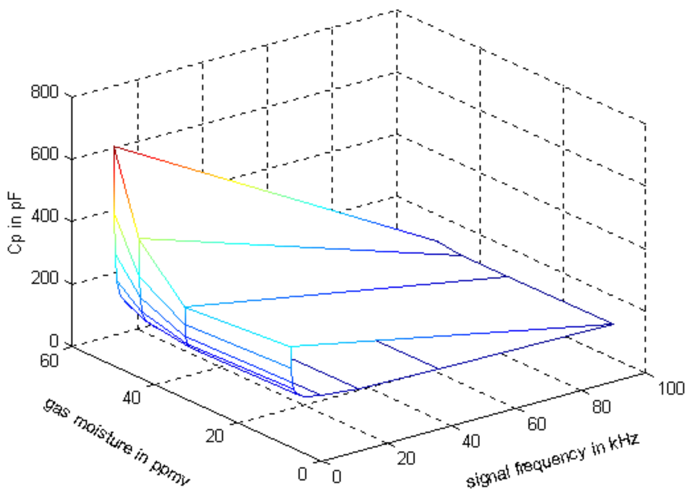


Fig. 4. Capacitance change of PS gas moisture sensor for the variation of both moisture content and signal frequency from 100 Hz to 100 kHz (Solatron Impedance analyzer, 1 V rms).

Fig. 5 shows the change in capacitance with moisture for the signal frequency of 464 Hz. Experiments are also conducted to see the change in conductivity with moisture for the signal frequency of 464 Hz and shown in Fig.7. Since capacitance is proportional to conductivity, thus capacitance increases with increase in moisture while resistance decreases with increase in moisture.

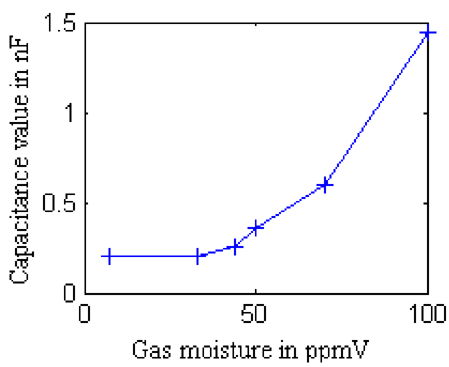


Fig. 5. Plot of capacitance change of porous silicon sensor at different moisture concentration of gases (ppmV) (Solatron Impedance analyzer, 464 Hz).

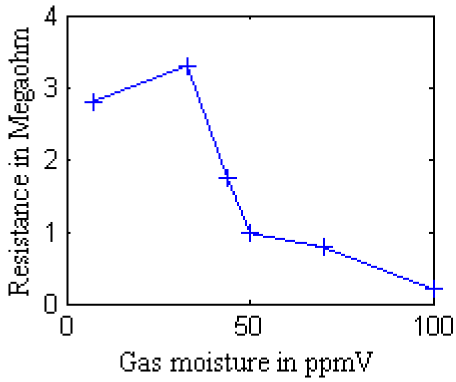
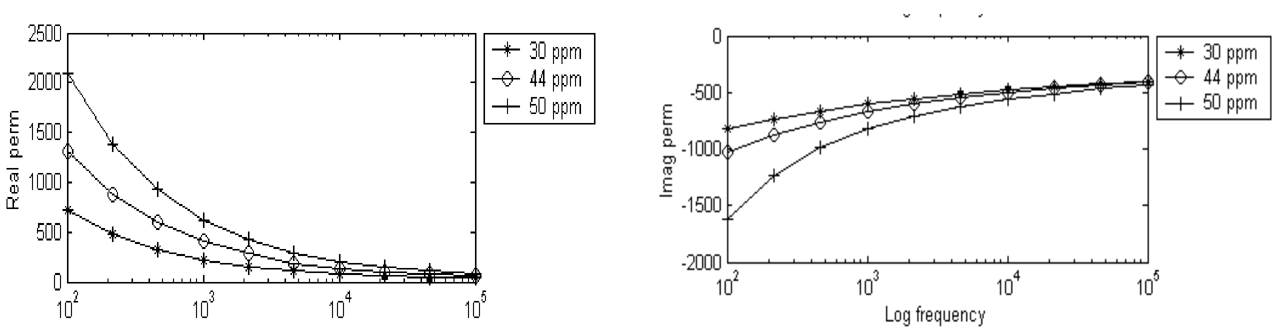


Fig. 6. Plot of resistance change of porous silicon sensor at different moisture concentration of gases (ppmV) (Solatron Impedance analyzer, 464 Hz).

5. Results and Discussion

It is evident clearly from these impedance spectra that the PS sensor is not really capacitive but resistance plays an important role in its operation. However, it is not advisable to develop sensor based on conductivity change because the effect of temperature for porous silicon conductivity sensor is more pronounced than capacitive sensor [1]. The experimental data are also analyzed to obtain the both real and imaginary components of complex dielectric constant. These results are plotted in Fig. 7. These plots show the variation of real and imaginary components of dielectric constant with the log of signal frequency for fixed vapor concentration. These two components play an important role to decide whether the sensor should be capacitive or conductive. We see the dielectric loss in all moisture range, the dielectric loss increase in the direction of higher frequency as moisture level increases. The dielectric loss represents the energy transformation resulting from the relaxation of the molecule polarization. When cluster of water vapor enter the pores the relaxation comes from the friction and collision resulting molecular re-orientation in an electric field. The dielectric loss is composed of polarization loss and conduction loss. It is also seen from Fig. 4 that change of capacitance with moisture at low frequency is more than at high frequency. Thus it is advisable to select lower excitation frequency for PS capacitive ppm level moisture sensor. The capacitance change is sufficiently nonlinear which can be taken care of by the signal processing circuit.



(a) (b)

Fig. 7. (a) Plot of real component; (b) imaginary component of complex dielectric constant with log of signal frequency.

6. Estimation of Moisture Concentration Using ANN

To estimate the moisture concentration, the sensor characteristic is function approximated using artificial neural network ANN [12-14]. ANN based polynomial function approximation is one of the widely used approaches to model the nonlinear behavior of sensor. The main advantages of ANN application related to curve fitting for nonlinear characteristics are extrapolation capabilities and the processing of multi parameter data of sensors. After successful training, neural network generally give lower error outside the calibration range than polynomial extrapolation. Results of ANN interpolation inside the calibration range are also better for the sensor characteristics, which is nonlinear. Only disadvantage of ANN approach over polynomial interpolation is the requirements of sufficient experimental data [13]. However, the hardware implementation of trained ANN model of sensor is easier than polynomial interpolation. It involves some multiplication, addition and thresholding operations. The present application suits ANN application for the estimation of moisture concentration. Here the PS sensor output is connected in cascade with the multilayer perceptron (MLP) structure of ANN. The input of the ANN is the output of PS sensor corresponding to moisture and the ANN output is the moisture content itself. Schematic of the ANN model is sown in Fig. 8. Thus overall transfer function of the sensor and neural network connected in cascade is unity.

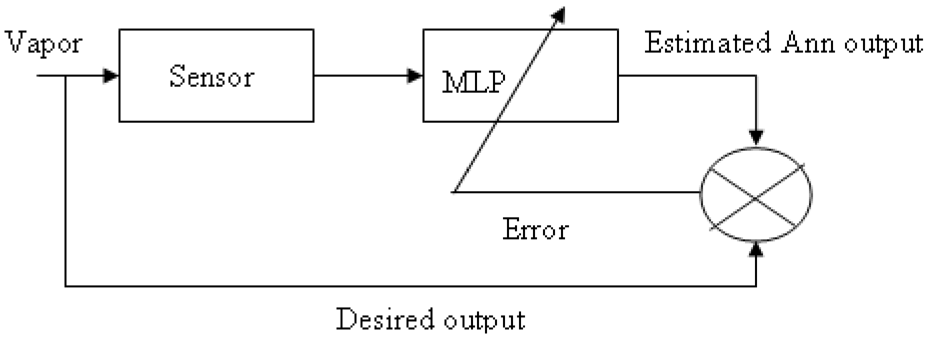


Fig. 8. Schematic of ANN model for moisture estimation.

The MLP neural network used widely for sensor function approximation consists of hidden layer neurons of sigmoid activation function that receives measured data and then transfers the output to another layer of linear neurons, which finally compute the network output by

$$N_{out} = purelin(U \times \tan sig(W \times x_i + B_1) + B_2), \quad (3)$$

where 'purelin' is the linear activation function for the neurons in the output layer and 'tansig' is the hyper tangent sigmoid activation function in the hidden layer. x_i is the input data, N_{out} is the neural network output and $B_1 \& B_2$ are the bias input. This architecture has proved capable of approximating any function with finite number of discontinuities of sensor [13]. The number of neurons in hidden layer depends on the non-linearity of sensor output characteristics. The best values of weights matrices; W, U and biases B can be computed by minimizing the mean square error (mse) defined by

$$mse(C_i, P_i) = \tfrac{1}{n}\sum_{i=1}^{n} N_{out}(C_i - P_i)^2, \quad (4)$$

where C_i is the desired moisture output of the i^{th} output neuron, P_i is the actual output of the i^{th} output neuron and n is the total neurons in the output layer. The training of the MLP structure is based on back propagation algorithm, which can minimize the error given in Eq. (4) [16]. During training a set of input values corresponding calibration points adjust the weights and biases of the neurons to minimize the mean squared error of the network.

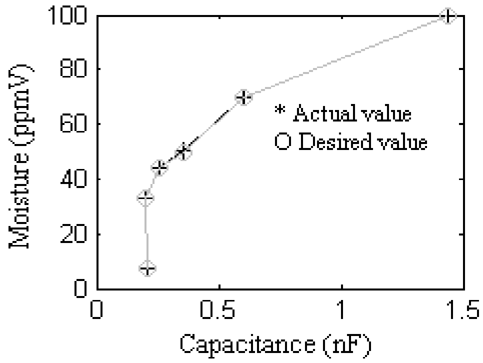


Fig. 9. Plot of estimated moisture output of ANN network and desired moisture output.

For our function approximation of sensor characteristic the optimum ANN structure is chosen heuristically. The chosen activation functions are the tan sigmoid in the hidden layer and linear in the output layer. The input layer has two neurons including bias and output has two neuron including bias but the neurons in the hidden layer is selected heuristically, which is 2-4-2. For training the MLP, the parameters used are learning rate = 0.1, mean square error = 0.00001 and maximum epoch of 300. Once the network is trained final weights and biases are stored for evaluation of the network and future estimation of moisture. The results of the function approximation are shown in Fig. 9. It shows estimated output of the ANN network. The actual output of the neural network and desired moisture level closely follows each other. Fig. 10 shows the percentage error in estimating the output from the ANN with the desired error. The error is only ± 0.3%. Thus multilayer perceptron based neural network can effective to estimate the moisture concentration from the trained weights stored in the memory of the computer.

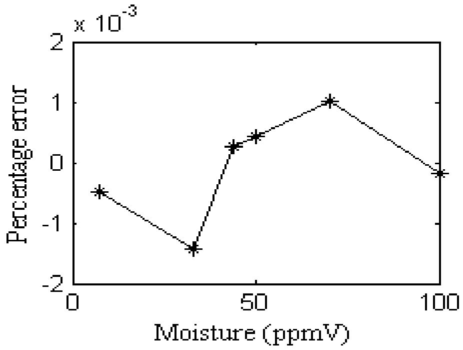


Fig. 10. Plot of percentage error between estimated and actual output.

7. Conclusion

The preliminary results discussed in this paper encourage the possibility of using porous silicon sensor for measuring gas moisture concentration in the ppm level. The measurement of medium and higher concentration humidity using PS is well established. However, the pore morphology and pore structure of the PS sensor in sensing ppm level moisture is not optimized. If pore structure is optimized the sensitivity of the sensor can further be improved. An ANN based approach is also discussed to approximate the functional relationship between input and output of PS capacitive ppmV moisture sensor. The ANN model of the sensor can directly measure the moisture concentration of the gas. The effectiveness of the ANN is well supported by the results.

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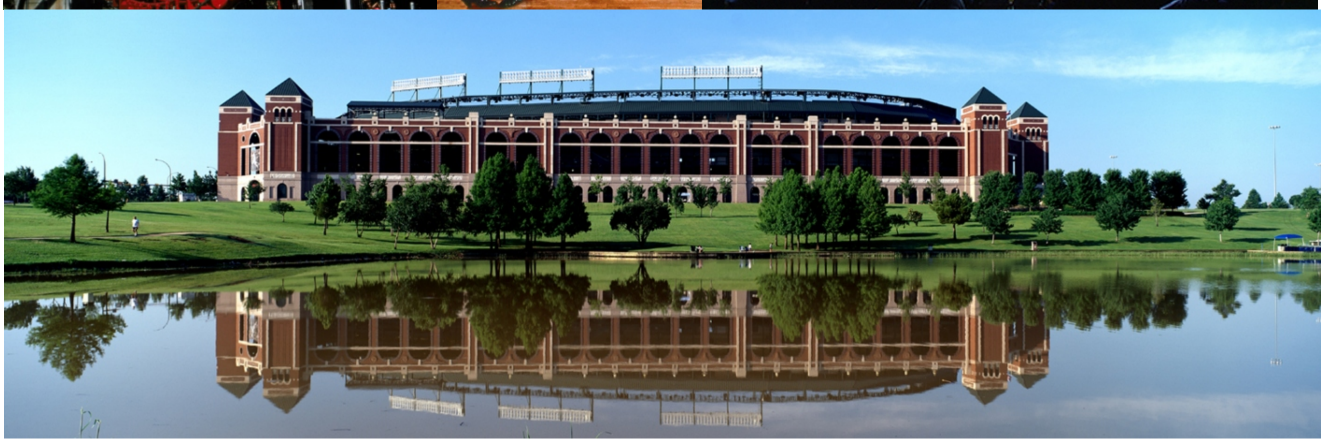

Guide for Contributors

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Sensors & Transducers Journal (ISSN 1726- 5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

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