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Application of Mathematical Model of Artificial Neural Network in PbO-doped SnO_2 Sensor for Detection of Methanol, CO and NO_2

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Abstract: In the present work thick film SnO_2 sensor was fabricated on a 1″x1″ alumina substrate. It consists of a gas sensitive layer (SnO_2) doped with PbO, a pair of electrodes underneath the gas sensing layer serving as a contact pad for sensor. Also a heater element on the backside of the substrate was printed. The sensitivity of sensor has been studied at different temperatures (150^0C-350^0C) upon exposure to methanol, CO and NO_2 vapour and gas and found maximum at 350^0C for Methanol. The structural analysis of the film was carried out by X-ray diffraction (XRD) pattern.

Keywords: Gas sensor, nanosized, PbO, SnO_2, thick film.

I. INTRODUCTION

Metal oxide semiconductor sensors based on electric conductivity measurement have been used extensively for gas detection. The sensing properties of the material are based on the adsorption of the gas molecules on it surface which produce changes in their conductivity [1]. The advantage of the SnO_2 thick film gas sensor is its high level of sensitivity, simple design, low weight and cost effectiveness. Also, these gas sensors could achieve more improved sensitivity to organic compounds by the control of particle size and addition of promoter [2,3]. Despite the high sensitivity, disadvantages such as lack of reproducibility, long term stability and selectivity for specific gases are frequently observed; moreover, the sensitivity to ambient moisture strongly interferes with the conduction mechanism. A better control of the number distribution and size of the grains and of the intergranular boundaries, together with the addition of catalysts and other promoters during preparation, can contribute in limiting the effect of the previously mentioned disadvantages [4].

Tin oxide layer used in the sensors is usually doped by other substances (Pd, PbO) as catalyst to improve the sensitivity and selectivity. It also can contribute to the generation of vacancies at lower temperatures. The chemical reaction provides oxygen vacancies which cause the change of electrical conductance of SnO_2 layer [5]. The effect of dopants also depend upon the processing method and target gas. The sensing properties of SnO_2 sensors (sensitivity, selectivity and reproducibility) depend on several factors, mainly grain size and specific surface area. Sensing mechanism of semiconducting gas sensor is based on the surface reaction of semi conducting oxide. In air, molecular oxygen is chemisorbed in the form of O_2^-, O^- or O_2^{2-} depending on operating temperature and deplete electron from the surface, leading to reduction of conductivity. Upon exposure of the reducing gas to the sensor surface, the chemisorbed oxygen reacts with the reducing gas and electrons are subsequently reintroduced into the conduction band, leading to enhanced conductivity [6]. It is believed that the exposure of gas causes, only the surface properties of the grain to change but for nanosized sensor, especially when crystal dimension approaches to thickness of charge depletion layer, the change in the properties of the whole grain, not just at the surface, is observed on the gas exposure.

1.1 Model of a Neuron

An artificial neuron is a unit having several weighted inputs and one output. Thus, we can say that a neuron is a function of two vector variables.

A neuron with k inputs transforming a set $X \subset \mathrm{IR}^k$ of input signals (a k-neuron on X) is a function

$$F : \mathbb{R}^k \times X \ni (\vec{w},\vec{x}) \longmapsto F(\vec{w},\vec{x}) = f(\langle \vec{w},\vec{x} \rangle) \in \mathbb{R},$$

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where wis a weights vector, $\langle\cdot,\cdot\rangle$ is a real scalar product, and $\mathbf{f} : \mathbb{R} \longrightarrow \mathbb{R}$ is called an activation function of the neuron. If f is a linear operator, then the neuron is called linear. A function is said to be a trained k-neuron on X.

$$F^* := F(\vec{w},\cdot) : X \ni \vec{x} \longmapsto F^*(\vec{x}) \in \mathbb{R},$$

1.2 Mathematical model of a multilayer ANN

The proposed model is based on graph theory. Let us recall its basic definitions.
An ordered pair (A,E), where A is a finite set of nodes and E $\subset A\times A$ is a set of oriented edges, is said to be an oriented graph (orgraph).
Set that $(a_i, a_j) \in E$ is the edge directed from the node ai to the node a_j.
The nodes set power of a graph G is said to be the degree of the graph G and will be denoted by δG.
Let a graph G = (A, E) be given. The power of the set {aj :(aj,ai) ∈ E } is said to be an input semidegree of the node ai and is denoted by $\delta+_{ai}$, whereas the power of the set $\{a_j : (a_i,a_j) \in E\}$ is called an output semidegree of the node ai and is denoted by $\delta-_{ai}$.
Orgraphs are often used in the definition of an ANN. The orgraph nodes are identified with neurons of an ANN, whereas directed edges determine inputs where the output signal from a given neuron is sent.
Assume that the following objects are given:
G:= (A,E) – an orgraph of a degree δ_G such that $\{a \in A : \delta_a+ = 0\}$ 6 ≠ ∅;
γ : A ∍ a → γ (a) ∈ $\{1,...,\delta_G\}$ – a bijection mapping;
F – the set of all neurons;
α : A ∍ a 7-→ α (a) ∈ F; if $\delta_a+ = 0$ then α(a) is a k-neuron, otherwise α(a) is a $\delta+_a$ -neuron;
W – a set indexing all inputs of neurons in the ANN;
β : E →W – a bijection mapping.

II. Experimental Work

Pure SnO_2 powder was prepared by reacting slowly $SnCl_4.5H_2O$ with ammonia water (NH_4OH). After some time the white precipitate of tin hydroxide was obtained which was washed with distilled water so as to remove excess ammonia chloride. Further, the precipitate was filtered and dried in an oven at about 150^0C. The powder thus obtained is the tin hydroxide which was calcined at 400^0C for four hours to get the tin oxide. Doped SnO^2 Powder was obtained by mechanically mixing the required amounts of PbO (1% by weight). This mixture was ball milled for 6 hours to get homogeneous powder and then sintered at 800^0C for 2 hours in a furnace. To get a proper paste, PbO-doped tin oxide powder was mixed with lead glass powder and the organic binder (ethyl cellulose) and ball milled for 4 hours. Then α-terpineol and Diethyl glycol monobutyl ether were added to the mixture and kept at 80^0C for 24 hours. Thick film of PbO-doped was prepared by screen printing technique onto alumina substrate as shown in fig.1.

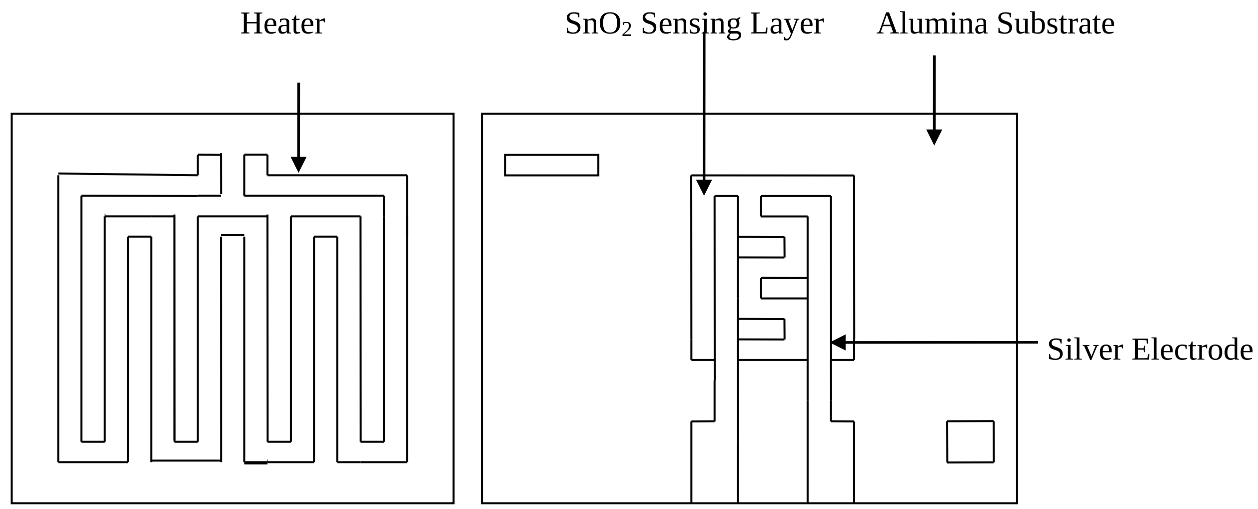

Fig.1. Schematic of fabricated sensors

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The phase identification of the powder was carried out by X-ray diffraction(XRD).The prepared sensor was exposed to the varying concentration of Methanol , CO and NO_2 in a locally developed test chamber having volume 2047 cm^3 kept at metal base. The change in resistance of sensor is measured using KEITHELY 195A multimeter. The testing was carried out in a test chamber [7] with a provision to inject the test gas. The Schematic Diagram of the measurement setup is shown in fig.2

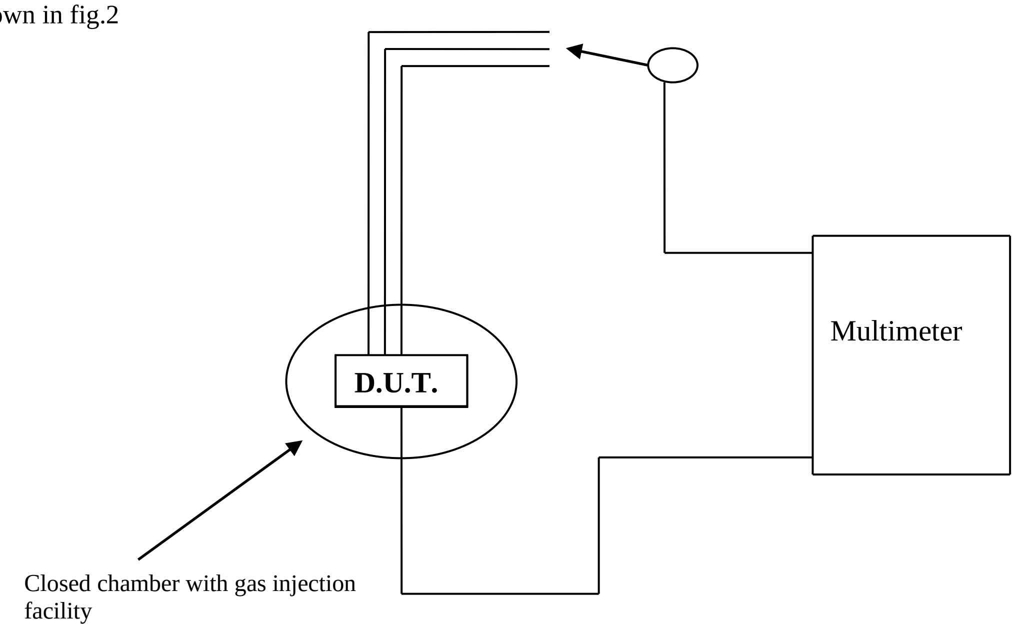

Fig. 2 Schematic diagram of measurement setup

III. RESULT AND DISCUSSION

3.1 Performance Characteristics of the Sensor

The sensitivity of fabricated sensor was studied at different fixed temperatures (150^0C-350^0C) with varying concentration of methanol, CO and NO_2 and found maximum at 350^0C which is shown in fig.3.

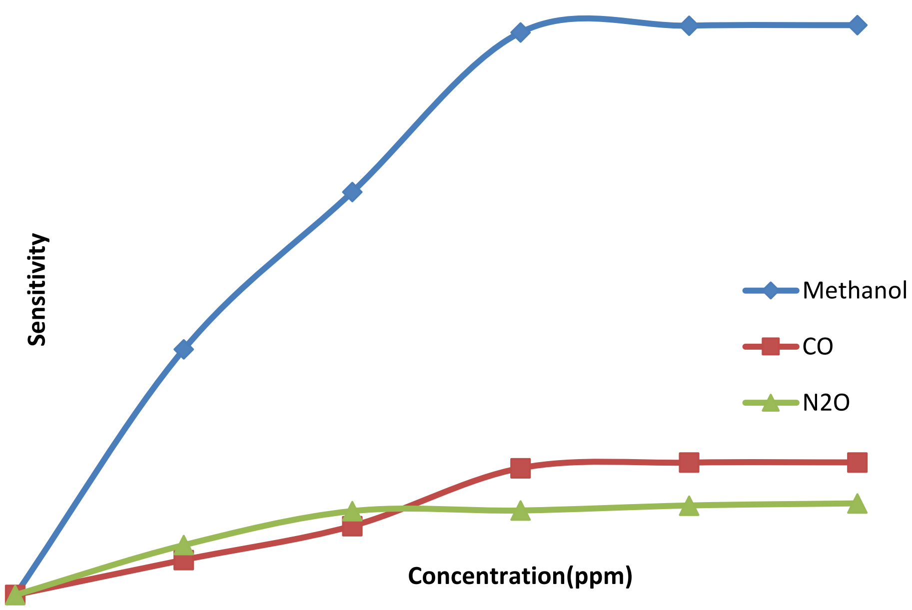

Fig.3 Response of SnO_2 sensors (1%, PbO doped) on exposure to methanol, CO and NO_2 at 350^0C

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It is evident from these results that sensitivity of sensor increases initially with increase in gas concentration and attains the saturation value after some time [8]. It can be inferred from these figures that PbO-doped sensor have the maximum sensitivity at 350^{0}C for methanol whereas for CO and NO_2 gas sensitivity was found to be minimum. Fig.4 shows the experimental plot of sensitivity versus operating temperature of methanol, CO and NO_2 at fixed concentration (500 ppm) for the sensor. It is clear from figures that sensor shows better sensitivity towards methanol than CO and NO_2 at 350^{0}C.

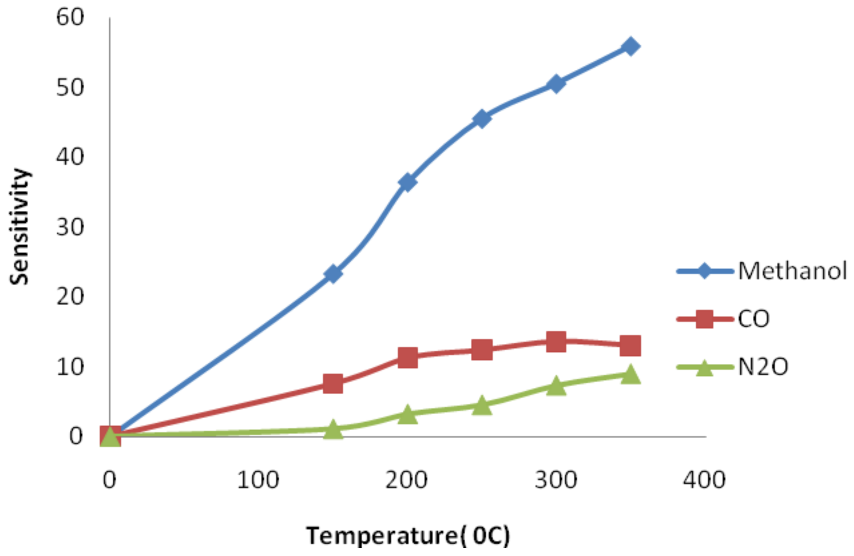

Fig.4 Variation of sensitivity of 1% PbO-doped SnO_2 sensors for Methanol, CO and NO_2 with operating temperature for constant concentration 500 ppm

Purelin transfer function retrieval capability of the 1 % PbO-doped SnO_2 based thick film gas sensor for methanol at 350°C. The validation performance goal was (0.011) achieved at zero epochs for purelin network transfer function network shown in Fig 5. The Gradient Descent Backpropagation with adaptive learning rate algorithm was used regression parameter of train data (0.97678) and output target train data (0.96097).

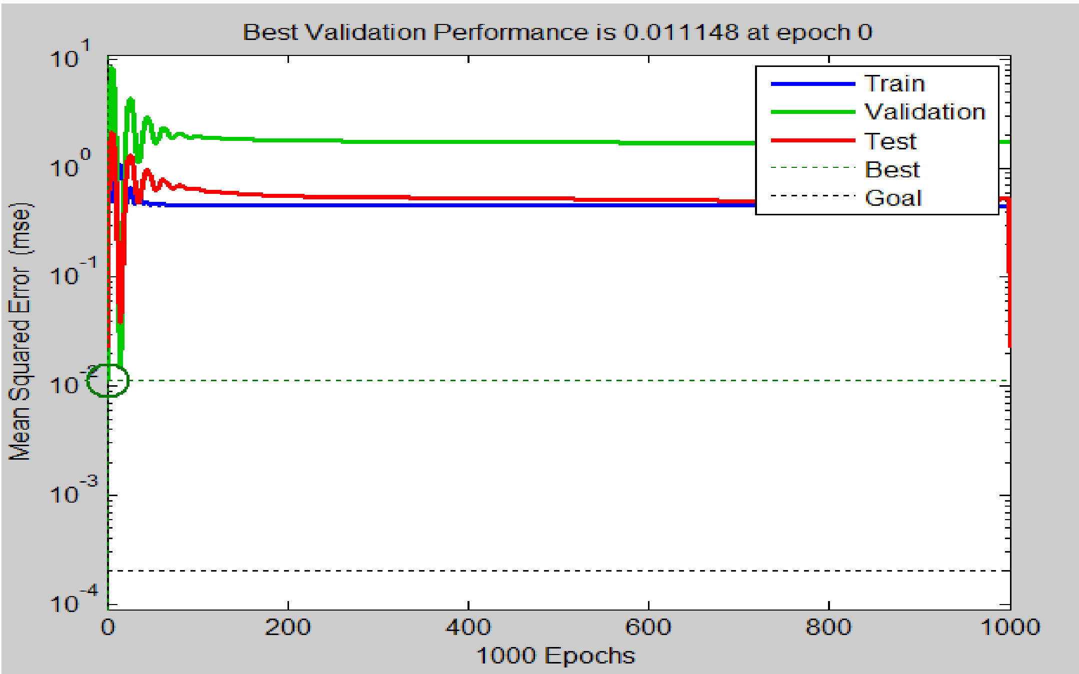

Figure 5. Results of validation performance in purelin network transfer function for Methanol

3.2 Structural analysis

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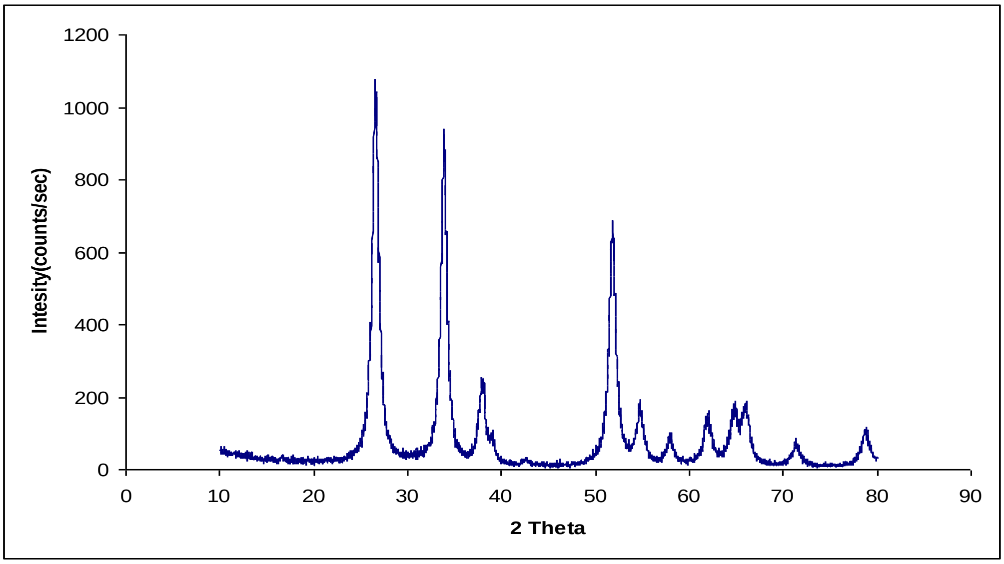

Fig.5 XRD Pattern of 1%PbO-doped SnO_2 sensor

The XRD pattern is shown in fig.5. XRD measurement was carried using an 18 kW rotating anode (CuKα) based Rigaku powder diffractometer. The observed pattern has several peaks for different angles. All the reflection peaks were indexed. The maximum of peak occurs at 26.43^0. The comparison of XRD pattern with standard chart reveals that all the peaks of the pattern correspond to different plane orientations of tin oxide, confirming the tetragonal structure with lattice constant a=4.7283, c=3.1714. The maxima occur for 110 hkl values. The XRD patterns confirms the almost 90% SnO_2 phase with tetragonal structure. The nature of the XRD pattern does not change on substitution of PbO in the host material. The absence of peaks of PbO in XRD may be due to small amount of PbO. The crystallite size found to be 20.45 nm, when calculated from X-ray diffraction pattern using Debye Scherrer formula [11]:

$$D = 0.9\frac{\lambda}{\beta}\cos\theta$$

Where D is the mean crystallite diameter, λ is the X-ray wavelength (1.54056Å), k is the Scherrer constant (0.90) and β the full width at half maximum (FWHM) of the diffraction lines.

IV. CONCLUSION

It is concluded that fabricated sensor doped with PbO shows better sensitivity towards methanol. PbO-doped SnO_2 sensor can detect methanol CO and NO_2 but only selective for methanol. The sensor shows maximum sensitivity at an operating temperature of 350^0C for all gases. The experimental result is also validated using Artificial Neural Network. The structure of the thick film sensor was analyzed through XRD. The XRD results showed that thick film was tetragonal crystal structure of SnO_2. The crystallite size was found to be 20.45 nm.

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BIOGRAPHY

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