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Synthesis and implementation of polycrystalline ferrite material for smart sensor module

S.N. Patil [a], J.D. Deshpande [a], S.K. Tilekar [b], B.P. Ladgaonkar [b], A.M. Pawar [a]

[a] Department of Electronics, Tuljaram Chaturchand College, Baramati, Dist: Pune 413 102, India
[b] Department of Electronics, Shankarrao Mohite Mahavidyalay, Akluj, Tal:Malshiras Dist, Solapur 413 101, India

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ABSTRACT

Keeping pace with facets of nanotechnology and its applications in the field of development of smart sensor module polycrystalline ferrite material plays significant role in development of sensing material. Hence, MgZn and NiZn polycrystalline ferrite have been synthesized by co-precipitation method. The formation of the materials is confirmed by characterization by X-ray powder diffraction, FTIR absorption. From the results of X-ray diffraction investigation, formation of single phase compositions is confirmed. The resulted structure is FCC with 311 as a prominent reflection. The average particle size obtains by using Scherrer relation and the range from 40 nm to 48 nm. The existence of the nanoparticles results into increase of the surface area required to favour adsorption mechanism. FTIR spectrographic studies, reveals the existence of various modes of the lattice vibration. These materials were used for development of the sensor for monitoring of Alcohol gas and temperature. The detection of alcohol and temperature is essential in various industries and for environmental prediction as well. The sensing elements of the sensors are developed using thick film technology and alcohol and temperature sensitive electrical properties are investigated. An operating temperature was optimized to 135 ^{0}C for gas sensing application. Deploying glass and ceramic substrate the sensor is developed. Electrical resistance of the sensor (R_g) is measured for alcohol gas and environment temperature and it shows decreasing trend with increase in the sensing parameters. Deploying this material smart electronic system is design using advance microcontroller. From, the results of this investigation, it can be concluded that, the compositions under investigation can be suitably used for deployment of Smart Sensor for sensing of alcohol and temperature.

Selection and Peer-review under responsibility of the scientific committee of the International Conference on Multifunctional and Hybrid Materials for Energy and Environment (MHMEE-2020).

1. Introduction

Now a day, development of smart sensor module for precise measurement and monitoring of environmental parameters, hazardous gases is need of hour for various applications. For smart sensor module sensor plays vital role. To develop sensor, sensing material plays significant role. On survey, it is found that monitoring of concentration various gases is need of hour, particularly for chemical industries, pharmaceutical, agricultural [1,2], food processing etc. To cater this need emphasizing an embedded technology smart sensor module with promisingly feature is required. Therefore, the research is going on in the field of development smart sensing material [3]. Different types of gas sensors have been developed wherein the materials such as zinc oxides, tin oxides, tungsten oxides etc. are used [4]. However, the ferrite materials are highly suitable for sensor based applications, due to its semiconducting behavior [5,6]. Therefore, the polycrystalline spinel ferrites of nano particlesize are synthesized using co precipitation method. On literature survey it is also found that, Polycrystalline ferrites exhibit interesting electrical properties, wherein the semiconducting behavior is realized. The electrical properties such as dc resistivity. These ferrite materials are highly resistive and its resistivity is mostly sensitive to the microstructure of the compositions. The ferrite materials of nanostructure exhibit nano particles and very small grains with uniform grain distribution. This leads to increase in the effective surface area. The gas sensing properties are mostly based on the surface phenomenon such as chemisorption and physisorption. The surface of ferrite materials at typical

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operating temperature reveals chemisorption of the oxygen species needed for physisorption of oxidizing or reducing gases [7]. Therefore, these materials are suitable for gas sensing applications. The exponential dependence of electrical conductivity of ferrites with temperature can be attributed to the semiconducting nature. In case of the ferrites the electrical conductivity is due to thermally activated charge carriers in the ionic lattice, which can be explained on the basis of hopping model [3,8]. Therefore, emphasizing the development sensor module, wherein embedded philosophy is realized, the polycrystalline ferrite based sensing materials are synthesized and studied for their gas sensing and temperature properties. The results of the investigation are interpreted in this section.

2. Experimental

The ferrite materials exhibit interesting properties, such as structural, electrical and magnetic. These intrinsic properties are strongly dependent on the chemical compositions, method of preparation, sintering time, sintering temperature of composition, particle size etc [8]. By considering and controlling these parameters, the compositions of the ferrites with chemical formula $Mg_xZn_{1-x}Fe_2O_4$ and $Ni_xZn_{1-x}Fe_2O_4$ (x = 0.2, 0.4, 0.6, and 0.8) have been synthesized by using co-precipitation method. For confirmation of structural details the compositions were characterized by standard tools like X-ray powder diffractometry and FTIR spectrophotometry. For application areas and nature of measur and sensor it is proposed to employ various shape, size, and substrate. Cylindrical glass substrate for sensor development. However, emphasizing the salient features needed for present embedded system design, it is proposed to deploy the glass and ceramic materials as substrate. X-ray diffractograms of compositions, $Ni_xZn_{1-x}Fe_2O_4$ and $Mg_xZn_{1-x}Fe_2O_4$ of polycrystalline ferrites, obtained from the diffractometer, and typically MgZn ferrite series depicted in Fig. 1. These diffractograms, show well defined reflections without any ambiguity. An intensity of 311 reflections is found maximum, supporting the fact that 311 reflections is predominant reflection.

3. Temperature dependant DC electrical resistivity Measurement:

To keep pace with the objectives of the present research work, the thick film sensor is design using ceramic and glass substrate. The ceramic materials such as Alumina are highly resistive ($\rho > 10^{14}\Omega$cm), highly rigid in nature and good thermal conductivity [SNP temp]. Therefore, these materials are mostly suitable as a substrate material for development of the thick film sensor and these sensor suitable for interfacing to electronics circuits. Therefore, by using screen painting technology the composition deposited on ceramic substrate as well as on glass substrate shown in Fig. 2 [7,8]. Glass substrate is suitable for gas sensor application to generate elevated temperature essential for gas sensing application. The silver paste bands are deposited on both ends of the film to establish the electrical contacts and this sensor is employed for further investigation of electrical properties. These properties are depends upon environmental parameters. Therefore, temperature dependent electrical resistivity properties were investigated. The electrical resistance of sensor measured with temperature range 30 ^{0}C to 150 ^{0}C and resistance against temperature graph shown in Fig. 3. On inspection of these graph, it is found that, the sensor showing negative temperature of coefficient of resistance and suitable for sensor based applications.

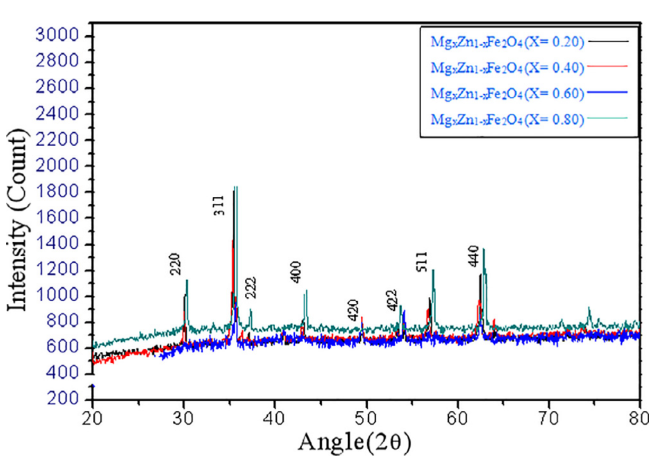

Fig. 1. X-ray diffractograms of compositions, $Mg_xZn_{1-x}Fe_2O_4$.

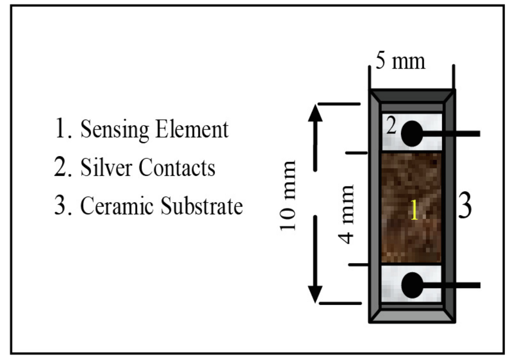

Fig. 2. The schematic of polycrystalline ferrites sensor developed on ceramic substrate.

The sensors for the compositions x = 0.20 and 0.80 show high resistance. Whereas, the resistance of the compositions for × = 0.40 and 0.60 is comparatively low. Therefore, the sensors developed by using ferrite compositions for × = 0.40 and 0.60 reveal its suitability as temperature sensor with the range from room temperature to about 150 °C. The relative variation in the resistance (ΔR), in percentage, of the sensors is calculated by using an equation.

$$\Delta R = \frac{(Ra - Rt)}{Ra} \times 100 \quad (1)$$

where, Ra is resistance at ambient temperature, Rt is temperature dependent resistance.

4. Alcohol gas sensitive electrical Properties:

Gas sensing properties of compositions were studied by measuring electrical resistance (R_g) of the sensing element by exposing to the gases. Alcohol is reducing gas and hence it resistance decrease with increasing the concentration of alcohol. The detection of alcohol is essential in pharmaceutical, chemical industries, sugar industries. Now a day, road police use for checking drunk case in driving time. Therefore, it is proposed to develop Smart Sensor Module for detection of alcohol gas, wherein the sensor, developed with use of thick film of ferrite materials, is deployed on cylindrical glass substrate. A heating Element (HH) specialy designed and installed along the axis of the sensor to provide temperature to realize the process of chemisorption [8]. Use of such sensing element helps to improve the response of the sensors to the various gases. In order to achieve Ohmic contacts the rings of silver paste are deposited at both sides of the thick film to ensure electrical contacts for interfacing to the Smart sensor module.

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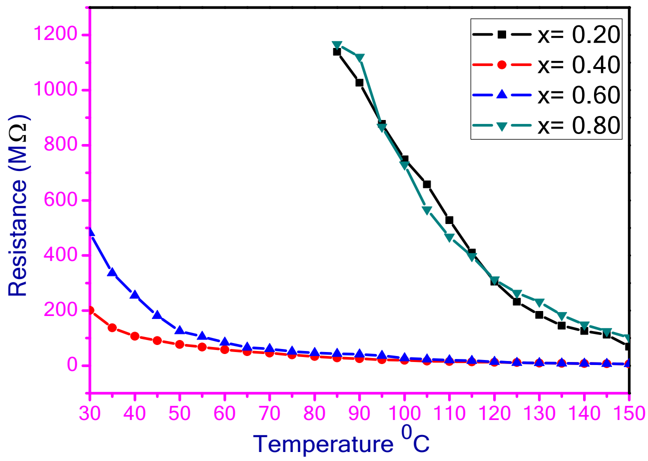

Fig. 3. Graph of resistance (R) against temperature (t) in ^{0}C for thick film of compositions $Mg_xZn_{1-x}Fe_2O_4$ deposited on ceramic substrate.

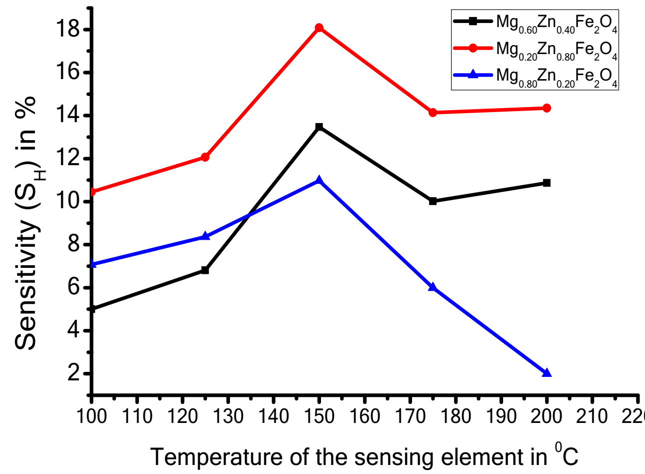

Fig. 4. Graph of sensitivity for S_{alch} gas against temperature of the sensing element in ^{0}C.

On literature survey it is found that, the semiconducting metal oxides, such as tin oxide, tungsten oxide, zinc oxide etc, are successfully proved as gas sensor materials. Gas sensing is the realization of surface phenomenon, wherein the phenomenon of chemisorption and physisorption is ensured. When the sensing element is heated to the sufficient temperature then the adsorption of the oxygen at the crystallographic sites of surface of the grains of polycrystalline material takes place. This is called as the chemisorption of the oxygen to form ionic species such as O^-, O_2^- and O^{2-}, which have acquired electrons from the conduction band of the surface material. Köseo_glu et al. [9] intensively studied the humidity sensing properties of MnNi ferrites prepared by chemical route using nitrides of the constituents. They reported that the $Mn_{0.2}Ni_{0.8}Fe_2O_4$ is sensitive for ethanol, acetone LPG and some oxidizing gases as well. Iftimie et al intensively studied the gas sensing properties of nanocrystalline MgMn ferrite [10,11]. They studied the response of MgMn ferrite for the gases such as LPG, ammonia, formaldehyde, ethyl alcohol, methane etc and reported that, the composition is most sensitive for LPG at temperature about 450 °C [10]. The gas sensing properties of polycrystalline Cu, Cd substituted Zn ferrites are investigated by Rezlescu et al. [12] and mention the sensitivity of the composition is temperature dependent. They reported the optimum sensitivity at temperature about 350 °C and at higher temperature. The operating temperature must be suitably low. Chapelle et al also studied the gas sensitive properties of copper ferrite for hydrogen gas and observed maximum sensitivity at 280 ^{0}C [89]. Gadkari et al. [13], investigated the gas sensing properties of the ferrites and reported that the composition of $Mg_{0.6}Cd_{0.4}Fe_2O_4$ exhibits about 78% sensitivity to the LPG gas at 225 °C [14]. Chromium substituted MgCd ferrite was investigated by Masti et al. [15] for their performance to the LPG Gas. They optimized the temperature required to complete the process of adsorption of oxygen at crystallite site by the phenomenon of chemisorption. The existence of porosity favours the process of adsorption [15,16].

Thus, for measurement of electrical resistance of sensing element it expose to the alcohol gas, the sensing material depicts the reduction in the resistance. This is the realization of conduction mechanism for n-type semiconducting metal oxides. The sensitivity (S_{alch}) in % of the materials for alcohol gas is given by the relation (2) and sensitivity against temperature graph shown in Fig. 4.

$$Salch = \frac{R_a - R_{alch}}{R_a} \times 100\% \tag{2}$$

where, Ra is the resistance of the sensing element at ambient condition and R_{alch} is the resistance of sensing element measured on exposing same to the alcohol gas. The values of Ra and R_{alch}, for all compositions under investigation, are measured by using highly precise digital multimeter model Tektronix Make model DMM4050. Inspection of these graphs, it is found that, sensitivity increases with increase in the temperature and become maximum at a typical temperature called operating temperature. At temperature range 145 ^{0}C sensitivity of $Mg_{0.20}Zn_{0.80}Fe_2O_4$ is high but ambient resistance is very high. As compare to $Mg_{0.40}Zn_{0.60}Fe_2O_4$ is less sensitive but low resistance, this parameter is consider for selection of sensor for designing sensor module.

5. Designing of smart sensor module

After successfully preparation of sensing material and sensing element, it is deploy for development of smart sensor module. Therefore, based on AVR Atmega 8 microcontroller based embedded system design for monitoring of concentration of gas and temperature measurement. To enhance the reliability and preciseness of the sensor module sensing material and sensor plays important role. The Smart Sensor Module designed to realize the plug and play philosophy [17–19]. Fig. 5 depicts the block diagram of Sensor module. After successfully design of hardware, Firmware is developed using CodeVision AVR, an IDE.

6. Result discussion and calibration of sensor node

6.1. Calibration for temperature

To display the values of the parameters in respective unit, calibrate the sensor module for display the temperature in ^{0}C and alco-

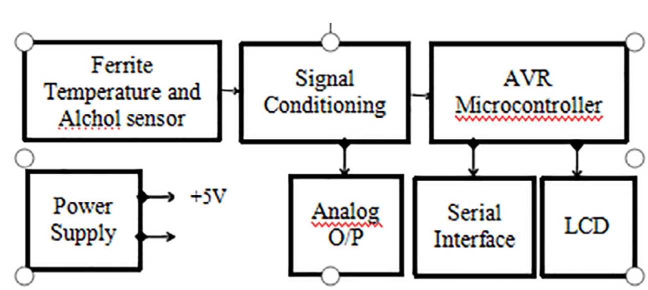

Fig. 5. Block diagram.

hol gas in percentage (%) unit. In the beginning temperature and gas dependent emf measure and plotted emf against temperature and gas concentration. An experimental set up is shown in Fig. 6. The temperature dependent emf is measure at output of signal conditioning stage and apply first analog channel of AVR. The graph is fitted by process of regression and the results into relations (1) and (2) . the graph is fitted in two regions, the first is from 30 ^{0}C to 60 ^{0}C and second regions is from temperature is from 60 to 100 ^{0}C were V_T is temperature dependent voltage shown in Fig. 7.

$$Temperature\ (t)^0C = ((V_T - 136.0)/9.335) \quad (3)$$

$$Temperature\ (t)^0C = ((V_T + 532.7)/20.83) \quad (4)$$

These equations, Eqs. (3) and (4), are employed in the firmware for calibration of the system to display temperature reading in ^{0}C scale on the display unit. The Table 1 depicts the observed temperature data on system and standard digital temperature meter with great reliability.

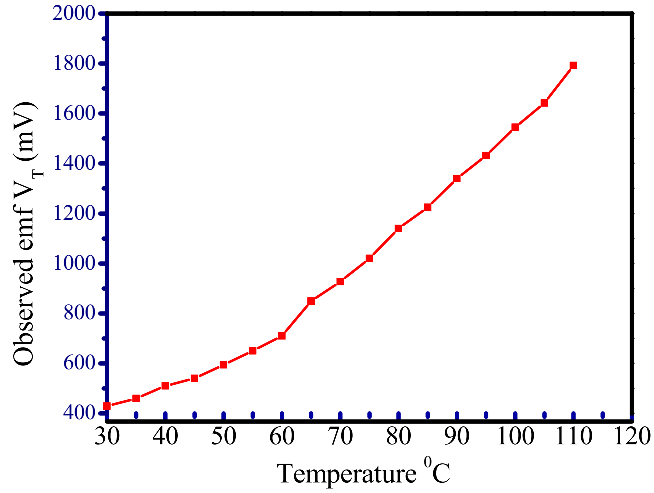

Fig. 7. The graph of observed emf (V_T) in mV against applied temperature in ^{o}C.

6.2. Calibration for gas sensor

Implementing the selection criteria, such as operating temperature, sensitivity, and ambient resistance etc, selected compositions of$Mg_{0.40}Zn_{0.60}Fe_2O_4$ sensing element deployed for development of present sensor module. It is known that, these sensors are resistive sensors and resistance sensor decreases increase in the concentration of the alcohol gas. The signal conditioning circuit provides proportional analog voltage in mV. This analog voltage (V_{G2}) is read into the AVR microcontroller for digitization and further processing. To achieve the output in scientific units, it has to be calibrated by employing standard means. The experimental arraignment shown in Fig. 8. For calibration standard alcohol of 10%, 20%, 50%, 80%, and 100% concentrate use and measure related voltage calibrate the system to display alcohol gas in % unit. These various % of alcohol available at alcohol plant of Sahakar Maharshi Shankarrao Mohite Patil Sahakari Sakhar Karkhana, Yeswantnager (Akluj). The concentration of alcohol dependent emf is measure at output of signal conditioning stage and apply second analog channel of AVR. The graph is fitted by process of regression and the results

Table 1
Temperature data shown by system under investigation and that obtained from standard digital meter.

Temperature shown by the Digtal temperature meter in ^{0}C	Temperature shown by the system under investigation (in ^{0}C)
28	28.1
30	30.1
35	35.0
40	40.0
45	45.01
50	50.3
55	55.3
60	60.2
65	65.3
70	70.0
75	75.2
80	80
95	95
100	100.1
105	105.01
110	109.96
115	115.02
120	120.2

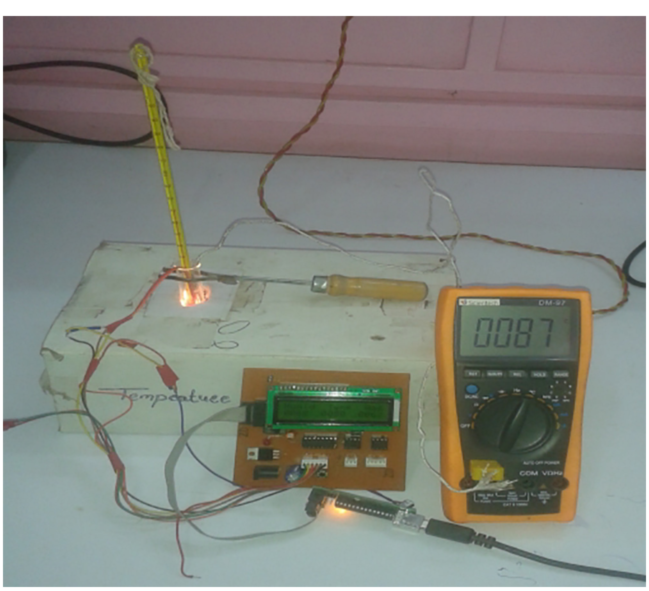

Fig. 6. Experimental arrangement for calibration of the sensor module for temperature.

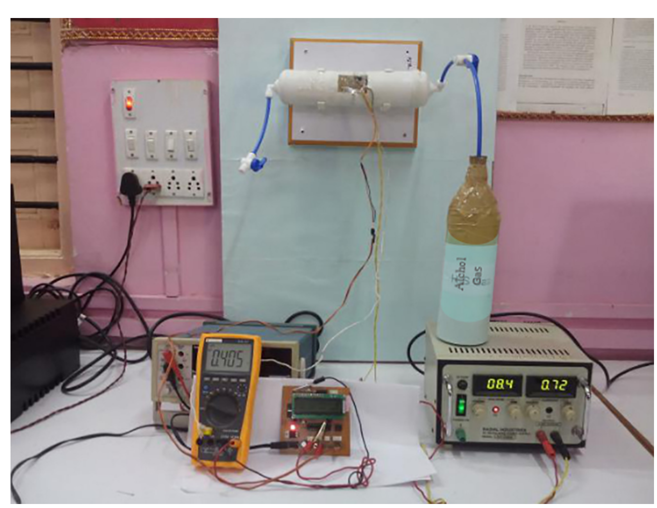

Fig. 8. Experimental arrangement for calibration of the sensor module for alcohol in %.

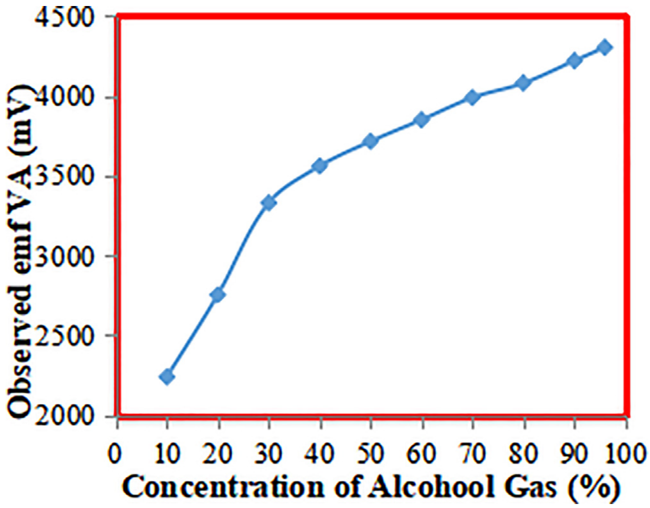

Fig. 9. Plot of observed emf in mV and concentration of alcohol gas in %.

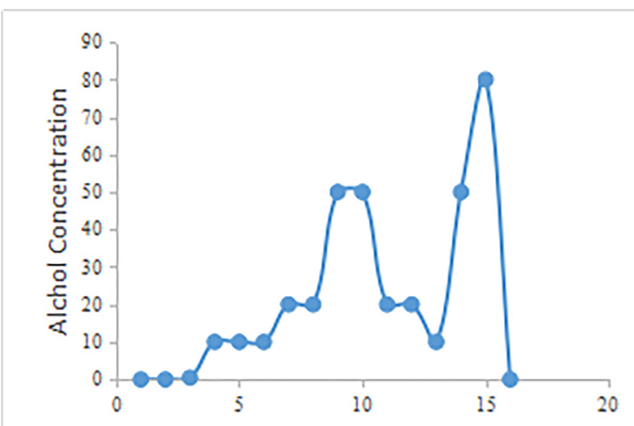

Fig. 10. Alcohol gas shown by the system under investigation (in %) at 147 ^{0}C.

into relations (4) and (5). the graph is fitted in two regions, the first is from 10% to 30% and second regions is from 30% to 100% were V_a is temperature dependent voltage shown in Fig. 9. Relation (5) and (6) put in software and measure different concentration of alcohol gas at 147 ^{0}C.

Concentration of Alcohol(in %)

$$V_a = 53.7V_{G2} + 1659 \tag{5}$$

$$V_a = 13.7V_{G2} + 2657 \tag{6}$$

Fig. 10 shows the response of system, when gas is present it shows in % otherwise display zero.

After successful calibration system is ready to measurement of temeprature in ^{0}C and alcohol in % unit. Indeed, to keep pace with the objectives, present work realizes the designing of smart sensor module for temperature and alcohol gas monitoring which depicts embedded technology, wherein both hardware and firmware are equally important. Thus, after calibration the designing process is completed the node is implemented on Sahkar Maharshi Shankarrao Mohite Patil Sunger Industries alcohol plant, Akluj Dist. Solapur and depicted in Fig. 11. As discussed earlier the samples available for calibration in alcohol plant. It is contributing significantly to the enhance pollution level of the areas close to industry area. Therefore, it is decided to implement the system in that region. On inspection of Fig. 11 the system is place near the alchol tank as well open area in industry area. Many workers working in that area as well as nearby the industry villagers leave. So it harm full to heath. The photograph depicts the implementation of nodes and data read for the system at 10.20 am to 3.45 pm on December. On inspection of Fig. 12, it is found that, the temperature of the environment was about 23 ^{0}C at the beginning of the experimentation. However, it is raised up to 26 ^{0}C and then reduced 3.00 pm. The concentration of alcohol gas is about 4–6% near by industry area. Mostly the alcohol concentration is maximum when valves changes open or close otherwise it is minimum in open air.

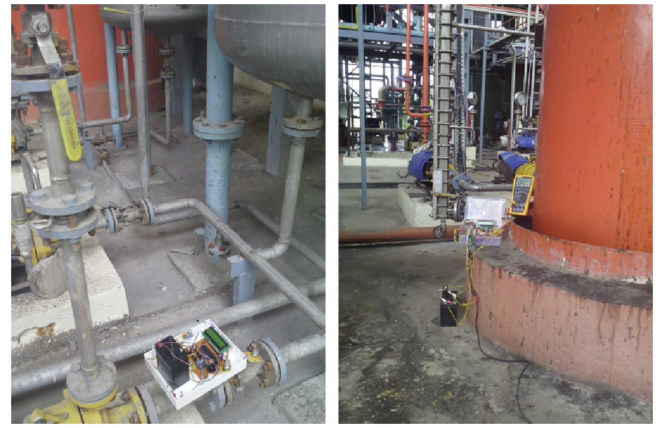

Fig. 11. Implementation of System.

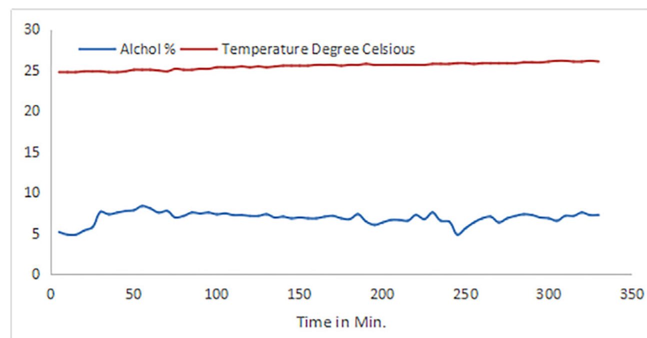

Fig. 12. The graph of temperature and alcohol gas for different times.

7. Conclusion:

The characterization of synthesized polycrystalline ferrite material reveals formation of single phase spinel compositions. The DC resistivity of the compositions under investigation shows negative temperature coefficient of resistance for the temperature and exposing gas concentration. However, near about room temperature, the variation in the DC resistance shows anomalous behaviour. Considering this property embedded system designed and calibrate for display temperature and alcohol gas concentration. On inspection of the data obtain from the system provide with high preciseness and system suitable for measurement of temperature and alcohol gas.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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