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Design and Construction of a Measurement Station for Electrical Characterization of Chemoresistive Gas Sensors

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Abstract:
A gas sensing measurement station consisting of mass flow control and mixing unit, heating and automatic temperature control unit, sensing chamber, resistance measurement and data logging unit was designed and constructed. The heating and automatic temperature control unit was made from 100 W, 220 V ceramic heater, type k thermocouple and REX-C100FK05-V*AN temperature controller. The unit maintains constant a temperature in the heating plate. The resistance measurement and data logging unit uses an Arduino Uno board to obtain the voltages across the device under test and a known resistor in a potential divider and the value of the known resistor to determine the resistance of the device under test. The measured resistance was logged to a spreadsheet in a computer system through PLX-DAQ data acquisition software. The measurement station was used to measure the resistance of some resistors and to test the behavior of MQ3 gas sensor in 205 ppm of ethanol in air and at 45℃. Results show that the gas sensing chamber was airtight and was able to maintain constant air pressure, the heating and temperature control unit was able to keep the temperature of the heating plate constant at preset temperatures between the room temperature and 900℃. The percentage error in the measured resistance by the resistance measurement and data logging unit was found to be 1.0% for resistance in the range 1 kΩ to 100 kΩ and 0.5% for resistance in the range 100 kΩ to 1 MΩ.

I. INTRODUCTION

Electrical resistance is an important property that depends largely on the composition of a material, its lattice structure, and geometry. For instance, the measurement of electrical resistance was used to study the damage in cement-based materials by Chung in 2003, The in situ moisture content of municipal solid waste was determined by Gawande *et al.*, in 2003 though measurement of electrical resistance. The technique was also used for detecting failure and damage in carbon fiber reinforced plastic (CFRP) materials by Kaddour *et al.*, in 1994, and Jie *et al.*, in 2011. Generally, resistance transducers are usually used to monitor physical quantities such as strain, humidity, and pressure.

The chemical nature of the surrounding medium of a material may affect its resistance. This principle is utilized in gas sensing systems, in which the presence of a particular gas is detected through the use of special chemical sensors called gas sensors. The resistance of a chemoresistive gas sensor varies with the concentration of a target gas. Therefore, accurate determination of the change in resistance of these sensors permits the measurement of change in concentration of the target gas. Consequently, the operation of a gas measurement system for characterizing chemoresistive gas sensors will depend mostly on the accuracy of its electrical resistance measurement unit.

A chemoresistive gas sensor utilizes a chemical reaction that occurs between a particular gas and a sensing material which results in a change in the resistance of the sensor, to determine the concentration of the gas. The use of germanium as a chemoresistive gas sensor was reported in 1953 by Brattain and Bardeen. Gas sensing abilities of semiconducting metal oxides was later reported by Heiland (1954), Bielanski et al. (1957), and Seiyama et al. (1962). Apart from semiconductor metal oxides, carbon nanotubes and conducting polymers in form of bulk and thin film or nanostructures are normally used as chemoresistive sensing materials. The sensing mechanism of semiconductor metal oxide gas sensors depends on the chemisorption of atmospheric oxygen at operating temperatures between 100 and 500 degrees Celsius. This leads to the formation of depletion and accumulation layer in n-type and p-type materials respectively. When the sensing material is exposed to the target gas, the gas molecules either donate electrons to or accept electrons from the valence bands in the material. This leads to change in the carrier concentration and consequently, the electrical conductivity of the material. Other types of gas sensor are calorimetric gas sensor, surface acoustic wave gas sensor (Showko and Jun, 2004), optical gas sensor (Robert, 2015), and electrochemical gas sensor (Matthew and Steven, 1983).

Synthesis and characterization of gas sensors is gaining attention of researchers nowadays due to the application of gas sensing in diagnosing health condition of humans through the exhaled air (Nasiri, 2019), monitoring or determination of air quality (Asthana, 2019), fire warning, and automotive application. To completely describe a sensor, its important properties that need to be measured include: sensitivity, stability, selectivity, accuracy, and recovery time.

A gas sensing station or set-up consist of basic elements, such as: gas sources, gas lines, flow meters, measuring chambers, devices measuring the sensor response, control systems, and equipment for safe discharge of gases. Other elements that ensure improvement in the accuracy of a set-up and increase the measuring capabilities are devices for stabilizing the measurement conditions in the measuring chamber, e.g. systems that control the relative humidity and temperature of the gas mixture in the measuring chamber, as well as the flow and concentrations of individual gases (Fu´snik *et al*, 2022).

In this paper, we describe the design and construction of a gas-sensing system in which the concentration of the target gas and temperature of the sensing material are controllable, the system is capable of measuring and determining the change in resistance of a gas sensing material in the presence of a target gas.

II. MATERIALS AND METHOD

The different units of the gas measurement station are: mass flow control and mixing unit, heating and automatic temperature control unit, sensing chamber and resistance measurement and data logging unit. The design and materials used in construction of these units are presented shortly.

A. Mass flow control and mixing unit

This unit consists of three manual mass flow controllers that can permit a maximum flow rate of 1.5 litres per minute (L/min) of oxygen, to control the flow of oxygen, nitrogen, and the target gas. A gas mixing manifold was fabricated, using steel pipe of radius 2 cm and length 10 cm. The manifold has three inlet ports and an outlet port. Each of the inlet ports of the mass flow controllers was connected to a gas bottle through a pressure tube, the outlet ports were connected to the inlet ports of the manifold. The outlet port of the manifold was connected through a pressure tube to the inlet port of the sensing chamber. Mixing of gases takes place in the manifold during dynamic gas testing.

B. Heating and automatic temperature control system

The heating unit was constructed using 100 W, 220 V ceramic heater and a steel block of dimension $7.5\text{ cm} \times 6.0\text{ cm} \times 1.0\text{ cm}$. The block serves as the heating plate. Two holes of diameters 0.8 cm and 0.6 cm were drilled into the block to accommodate the ceramic heater and a thermocouple. A ceramic chamber was fabricated to house the steel block such that only one of the large surfaces is exposed. The ceramic chamber prevents heat loss from the block to the surroundings through the other surfaces of the block. The temperature control unit was constructed using REX C100 FK05-V*AN temperature controller and a solid state relay SSR 40 DA. The unit uses type k thermocouple and it is capable of measuring temperature from 0 ℃ to 1000℃.

C. Gas sensing chamber

The gas sensing chamber was fabricated using 2.0 mm stainless steel plate. The plate was formed into a cylinder of 12.0 cm high and 20.0 cm in diameter. The bottom of the chamber was closed. Inlet and outlet ports were made with cylindrical pipes of diameter 3.0 mm, they were fitted with valves for regulating the flow of gas to and from the chamber respectively. The cover of the sensing chamber was made of a steel plate and a pressure gauge was fitted into its center. Flanges were made available at the top of the sensing chamber, around the outer circumference for securing the cover of the chamber with bolts and nuts during use. The region below the cover and the chamber was made airtight through the use of a gasket made of silicone. The chamber houses the heating plate with a thermocouple as well as the electrical measurement probes. Terminals for connection of wires to units in the chamber were provided. Fig.1 shows the temperature control unit, sensing chamber, and heating unit.

D. Resistance measurement system

Materials used in the system are: Arduino Uno board, four-channel relay module, 1 % tolerance resistors, multiturn variable resistors, and push-to-make (button) switches. The resistance measurement system was based on potentiometric method of comparing the resistance of the device under test (DUT) and a standard resistor (current shunt). Measurement of the voltage applied across the potential divider and the DUT was done with the Arduino

Fig. 1 (a) Temperature control unit, (b) Aerial view of sensing chamber showing the heating plate and connecting wires, (c) Lateral view of the chamber, (d) Closed sensing chamber, and (e) The heating unit.

board and the resistance of the DUT was calculated based on this measurement. The DUT and a resistor of fixed value, the current shunt, R_s, were connected to form a potential difference across a reference voltage, V_{REF}, as shown in Fig. 2(a).

The voltage, V_o across the current shunt, R_s is V_o. The voltage across DUT is $V_{REF} - V_o$, and the ratio of R_{DUT} to R_s is:

$$\frac{R_{DUT}}{R_s} = \frac{V_{REF}-V_o}{V_o} \qquad 1$$

The resistance of DUT can therefore be calculated as:

$$R_{DUT} = \frac{V_{REF}-V_o}{V_o} R_s \qquad 2$$

An external reference voltage, V_{REF}, was obtained from the 5 V pin of the Arduino, V_{REF} and V_o were both measured with the Arduino board. The board also performed the mathematical operation for calculating R_{DUT} according to Equation 2.

The 10-bit analogue-to-digital converter (ADC) in the Arduino board can provide a total of 2^{10} counts (0 inclusive), thus the board measures V_{REF} as 1023. When the resistance of the DUT varies between $0.1R_s$ and $10R_s$, the measure output voltage, V_o, would vary from 930 to 93.

Since the relationship between R_{DUT} and V_o is non-linear, V_o becomes very small at large values of R_{DUT} thus the change in V_o under this condition may not be accurately measured. For this reason, the minimum value of R_{DUT} to be measured was made to be $0.1R_s$ and its maximum value was limited to R_s so that V_o as will be measured by the microcontroller can vary between 930 and 511. Three current shunts, R_s, 10 kΩ, 100 kΩ, and 10 MΩ were used in order to make the system capable of measuring resistance from 1 kΩ to 50 MΩ. The current shunts were constructed from fixed resistance with 1 % tolerance and trimmers connected in series with each. The resistance of the combination was adjusted to the required value and it measured with Owon electronic digital multimeter, model XDM 1041.

The logging of the calculated resistance and time to a computer system was done by the Arduino board, together with a data acquisition software, PLX DAQ. The result of calculation was transmitted through the data acquisition software to Microsoft excel spreadsheet every second. The plot of resistance versus time was done by the computer through the spreadsheet for visual observation. The complete circuit diagram of the system is shown in Fig. 2(b).

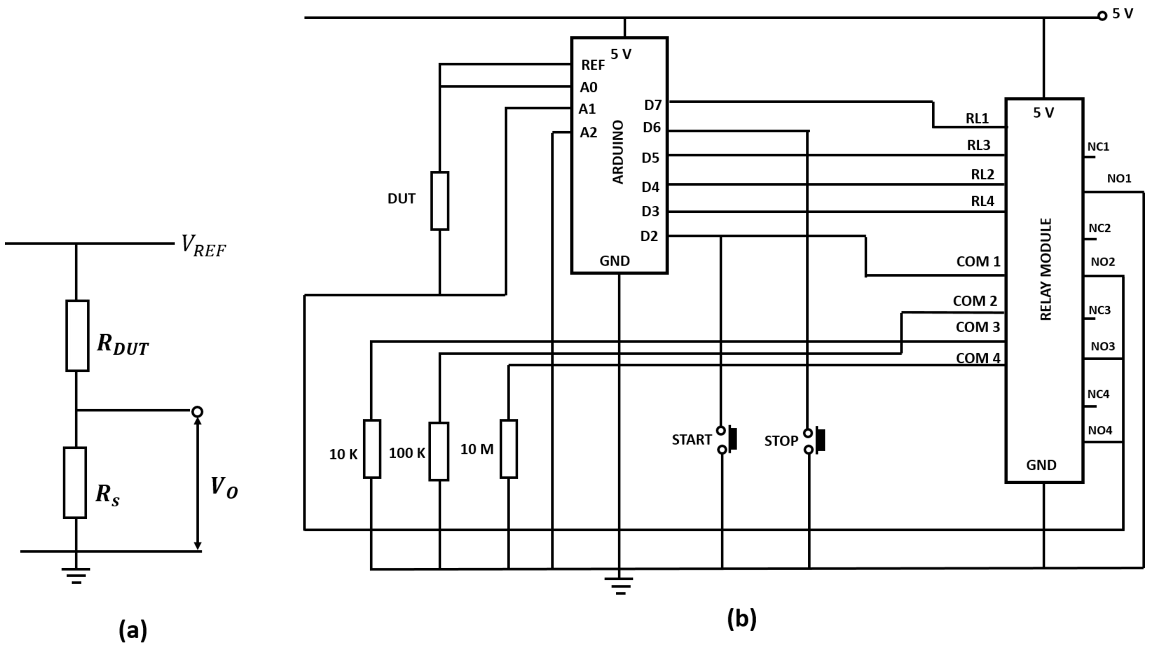

Fig. 2 (a) Potential divider arrangement for determination of resistance of DUT, (b) Circuit diagram of the resistance measuring unit.

E. *Operation of the circuit*

Initially, pins D2 and D7 of the Arduino board are at logic 1, other digital pins are in states that de-energize the relays. The circuit performs no operation under this condition. When the start switch is pressed, the logic state of the pin D2 becomes 0, this makes pins 4 and 6 to be at logic 1 thereby energizing relays RL1 and RL2 respectively. This selects the 100 kΩ current shunt and permanently applies logic 0 to pin 2 thereby making the microcontroller to run the program that calculates the resistance of the DUT. The microcontroller measures the voltage across DUT and the current shunt. If the measured voltages lie within the expected range according to the explanation in section D above, the microcontroller calculates R_{DUT}. It performs the operation ten times every second, calculates the average, and displays the result in a Microsoft excel spreadsheet.

However, if the measured voltages are outside the range, appropriate current shunt is selected and measurement repeated before calculating R_{DUT}. When the stop switch is pressed during the operation of the circuit, the microcontroller terminates the operation.

F. *Flow chart of the system*

A flow chart that describes the program on which the microcontroller operates is shown in Fig. 3.

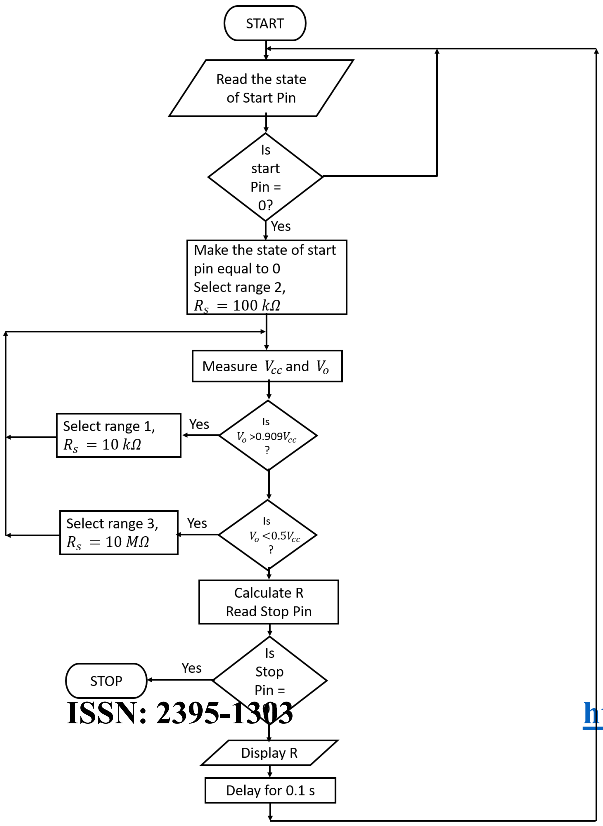

Fig. 3 Flow chart of the system

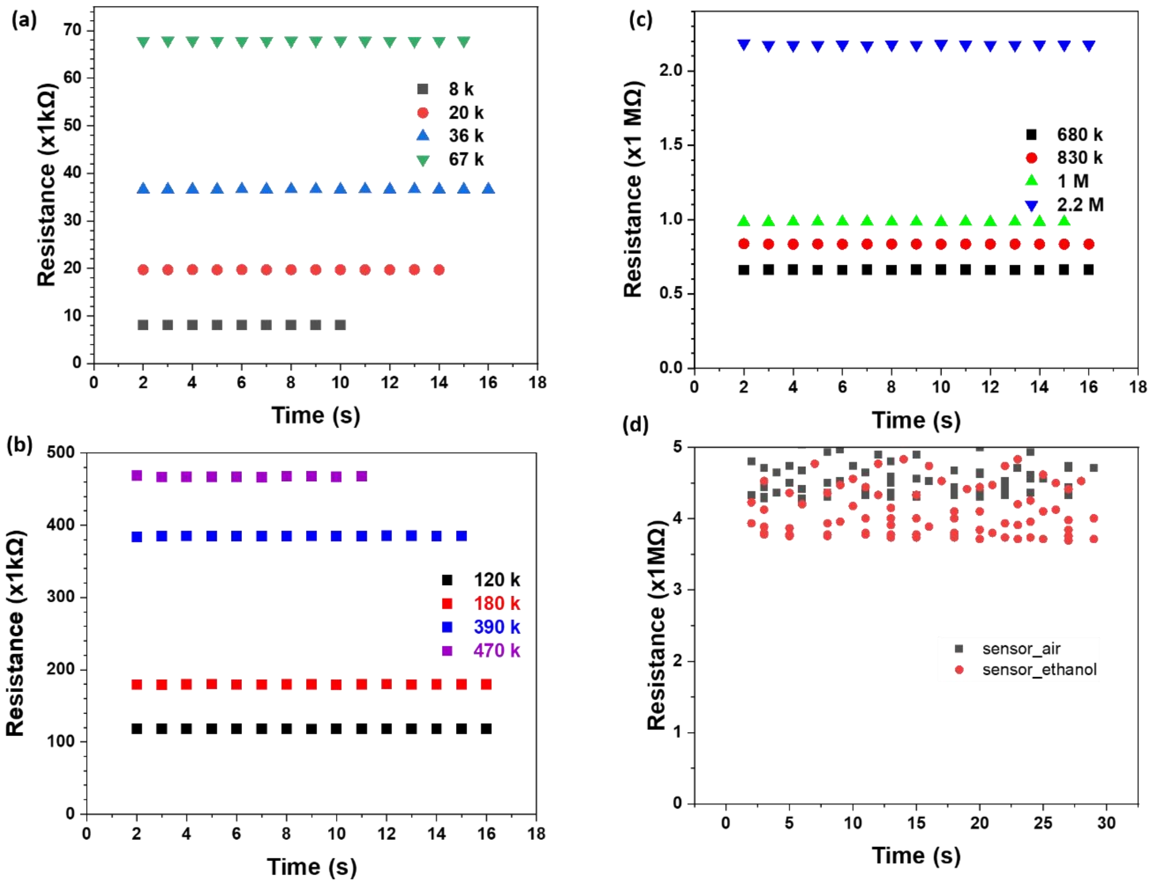

Fig. 4 Graphs of resistance versus time for the measurement of resistance in (a) 0 to 100 kΩ, (b) 100 kΩ to 1 MΩ. (c) 1 MΩ to 10 MΩ range respectively, (d) Graph of resistance versus time for the measurement of resistance of MQ3 gas sensor in air and in 205 ppm 0f ethanol in air.

III. TESTING AND CALIBRATION OF THE SYSTEM

Twelve resistors, 8.2 kΩ, 20 kΩ, 36 kΩ, 67 kΩ, 120 kΩ, 180 kΩ, 390 kΩ, 470 kΩ, 680 kΩ, 830 kΩ,1 MΩ, and 2.2 MΩ, were selected for the testing. The resistance of each resistor was first measured using the Owon electronic digital multimeter, each resistance was later measured with the electronic system for about 20 seconds. Results of the measurement was logged to a spreadsheet and the graph of resistance versus time was obtained in real-time.

The sensor in MQ3 gas sensor was removed from its module, the heater circuit was disconnected. The sensor was placed inside the gas sensing chamber and its resistance terminals were connected appropriately. The temperature of the heating plate was set to 45 ℃. The resistance of the sensor was measured for a few seconds when the temperature reached the set value. The measurement was repeated after 1 ml of 98% ethanol was introduced into a container in the chamber through a syringe, needle and a fine capillary tube. The concentration of ethanol air under this condition is estimated at 205 ppm. The measurement of resistance continues for about 200 s.

IV. RESULTS AND DISCUSSION

Graphs of resistance versus time for measurement in the range 0 to 100 kΩ, 100 kΩ to 1 MΩ, and 1 MΩ to 10 MΩ are shown in Fig. 4(a), (b), and (c) respectively. Fig. 4(d) shows the graph of resistance of MQ3 gas sensor versus time for measurement that was done in air and in 205 ppm of ethanol.

The resistance of the resistors as measured using with the electronic multimeter were 8.1 kΩ, 19.5 kΩ, 36.2 kΩ, 67.4 kΩ, 117.7 kΩ, 178.5 kΩ, 382.5 kΩ, 465.0 kΩ, 0.66 MΩ, 0.84 MΩ, 0.99 MΩ, and 2.19 MΩ respectively. The average reading of the measuring system for each of the resistors was 8.1 kΩ, 19.7 kΩ, 36.6 kΩ, 67.9 kΩ, 118.3 kΩ, 179.5 kΩ, 384.8 kΩ, 467.2 kΩ, 0.66 MΩ, 0.84 MΩ, 0.98 MΩ, and 2.18 MΩ respectively.

The percentage error was found to be 1.0% for measurement of resistance in the range 1 kΩ to 100 kΩ and 0.5% for measurement in the range 100 kΩ to 1 MΩ. It can be seen that the meter reading and the average reading of the system was found to be close when the resistance of the device under test was less than the resistance of the current shunt. An error of 0.5% was observed when the resistance of DUT lies between one and five times the resistance of current shunt. The graphs show that the average resistance measured by the system was close to the nominal value. The reading of the system when the gas sensor was tested in the chamber was erratic. However, the reading was found to have a minimum reading of about 4.5 MΩ when the sensor was in air and about 3.5 MΩ when the concentration of ethanol in the chamber was 205 ppm. The reason for this erratic behavior can be attributed to the fact that the sensor could not maintain a physical contact with the heating plate, thereby making it difficult for the sensing element to be at the required operating temperature.

V. CONCLUSION

Measurement of resistance between 10 kΩ and 2.2 MΩ with the system was successful and the error in the measurement was minimal. The logging of measurement data and real time plotting of graph of resistance versus time by the system was good. The temperature control of the heating unit was accurate, it controlled the temperature without overshooting. It is expected that the system will measure resistance of any device in form of pellets, thin film, or sheet as long as it can lie flat on the heating plate and maintain perfect contact with it throughout the period of measurement.

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