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Improving interoperability of catalytic sensors

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ABSTRACT

A huge number of combustible gas leaks occur every year at industrial and home facilities. These leaks may have grave consequences for health, environment and property. In this work we demonstrate the approach for improving the interoperability of catalytic sensors which are widely used in industrial monitoring applications for combustible gas leaks. To ensure methane detection within the broad concentration range (100 ppm–100% vol.), we propose using of two sensors with catalysts in the modified Wheatstone sensing circuit and to cover them with sealed caps with different holes to enable gas diffusion control.

1. Introduction

There is no accurate statistics on how many gas leaks are registered at industrial plants every year. Either detected or not, these leaks are not necessarily reported on, save for the cases when they result in serious damages or accidents. However, even negligible concentration of continuous gas leaks might cause a number of occupational diseases of the employees in gas industry. Thus, for instance, the factory workers dealing with methane suffer from the problems associated with the vegetative nervous system, e.g. hypotension, positive Aschner reflex. According to the Environmental Protection Agency (EPA) reports, in the United States alone the plants using methane emit nearly one billion cubic meters of this gas in the atmosphere. Approximately 80% of these losses occur due to leaky compressors, seals, valves, and connectors [1].

At present two general technologies are used for gas leak detection at industrial facilities. These are catalytic beads and infrared (IR) absorption [2]. Both of the techniques require that a network of wired detectors be deployed all over the territory to be monitored.

As recent research works demonstrate, interest to gas monitoring [3–5] and deployment [6] has noticeably increased. Several approaches have been proposed by the research community for gas detection so far. One of the solutions suggests using the energy-aware film (colorimetric) gas sensors [7,8]. It ensures low power consumption of the order of 3 mW, but long sensor response time (up to 5 min), which goes beyond safety requirements [9]. The gas platforms on the basis of laser spectroscopic trace-gas sensors [10] are, on the contrary, more power-hungry and faster in terms of response time. They are able to detect and quantify a variety of gas mixtures at part-per-million to part-per-billion (ppm-ppb) concentrations. However, such platforms have their disadvantages having power consumption at a level of 500 mA. Metal-Oxide (MOX) and electrochemical [11] sensors, like spectroscopic sensors in the above-mentioned approach, operate effectively in ppm concentrations, but cannot be used for detecting combustible gas leakage in the % vol. concentrations because of the risk of poisoning. Another approach to gas leak and potential fire detections which has become popular is video monitoring with cameras [12]. The cameras (usually mounted at the pre-set height and quite often rotating to cover the entire area of interest) take snapshots of the environment and analyse the obtained images to identify gas leaks. This approach requires sophisticated video processing algorithms and is characterized by high power consumptions which is a major problem for the autonomous monitoring systems, e.g. wireless sensor networks (WSN) [6,21].

In our opinion, the compromise solution which is able to secure both the long-term functioning of the sensors and their performance is the application of catalytic sensors. Catalytic sensors are widely used for detecting combustible and hazardous gases in the atmosphere. However, there is a number of reasons why they are

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not employed for the detection of gas concentrations in the full concentration range, i.e. from low and up to 100% vol. gas concentrations in the atmosphere. These reasons are:

- Poisoning of sensors by catalyst poisons;
- Sensor carbonization at high gas concentration in the atmosphere;
- Impossibility to detect low gas concentrations due to low response signal and high level of interference caused by, e.g. uncontrollable environmental parameters;
- Relatively short period of operation due to natural aging;
- Relatively high power consumption (150–300 mW per sensor [13]) which makes the sensors exploitation in WSN applications difficult.

Some of the above mentioned problems have been addressed recently [14–16]. For example, in order to reduce the power consumption of a sensor, the one sensor based sensing circuit [3] has been proposed instead of the widely used Wheatstone circuit. Extra reduction of energy consumption can be achieved by the fabrication of catalytic sensor using planar technology on anodic alumina membranes [17]. In addition, some more energy efficient measurement procedures [13] have been introduced.

There is a number of techniques aimed to compensate the environmental conditions for the measurement procedure have been proposed. The solutions proposed in [4] and [18] for temperature compensation are the modifications of the well-known Wheatstone circuit. Okazaki et al. [18] have proposed a temperature compensation method for the sensing circuit with active and reference catalytic sensors. The core idea of this method is that the sensors ought to be heated up to different temperatures with different heating currents. The heating of the active sensor is carried out to above thermal decomposition temperature, while the reference sensor is heated to below thermal decomposition temperature. The method secures long term stability, though not ensuring good selectivity, which sets limits to its application. To provide the precise measurement of the ambient temperature variation, an extra temperature sensor incorporated in a gas sensor has been proposed in [4]. Unlike the heater resistance measurement, this approach makes it possible to significantly improve the gas sensor stability. It should be noted, however, that integration of the temperature sensor adds to both complexity and cost. It also requires extra calibration for the temperature sensor (in addition to the gas sensor calibration). A much simpler approach based on the software algorithm for thermo compensation is proposed in [22]. The core of the measurement procedure is the four stage heating profile which enables low power consumption of sensing circuit and thermo compensation adjustment. The thermo compensation algorithm helps avoid the effect of the environmental temperature on the measurements by keeping stable zero-offset within ± 1 mV and ensuring low absolute error within 0.1% vol. The compensation for humidity is proposed in [19] by the application of stabilized nanoparticles as catalytic layer. The authors report on a short response time around 150 ms and good sensitivity ensured by the approach.

In the present work, we address the problem of the catalytic sensor application in the scenarios when low and/or high gas concentrations have to be detected. Our goal is to develop the techniques enabling the application of catalytic sensors for gas leaks monitoring in ambient conditions.

It is well known that catalytic sensors effectively operate in the 0.1–10% vol. range. At the lower concentrations the sensitivity of catalytic sensors degrades due to the lack of methane in the atmosphere, i.e. at the lower concentrations the methane burned on the heater of the sensor does not provide the significant conductivity change which could be measured. And vice versa: at the higher concentrations the burning process stops due to the lack of oxygen. Furthermore, when oxygen deficiency occurs, this leads to the reduction of sensitivity (response) of the sensor. The *novelty* of our work is in demonstrating the approach capable of improving the interoperability of catalytic sensors, i.e. the ability of their operation in the 100 ppm–100% vol. range. This interoperability is achieved by the application of a new measurement circuit and a mechanism for gas diffusion control which allows the exploitation of the same catalytic sensors for detecting both low and high concentrations.

This paper is organized as follows. We describe the problem we are addressing in this paper in Section 2. Our approach together with the experimental results and discussion is presented in Section 3. Finally, we provide concluding remarks and discuss our future work in Section 4.

2. Problem formulation

The reliability of aerological stations and sensor networks could be significantly improved by addressing the problem of sensors interoperability, i.e. when a sensor can successfully detect the gas concentrations in 0–100% range. This can be done by improving the "informativeness" of the sensors which employ the catalytic principle of operation towards detecting low concentrations defined by sanitary standards, as well as with high concentrations up to 100%.

Nowadays, low gas concentrations are detected using IR and semiconductor sensors. IR sensors are sensitive, expensive, and bulky. Their sensitivity can significantly decrease due to moisture condensation on the optical path at high humidity conditions when, for instance, the ambient temperature changes rapidly. Semiconductor sensors have another important disadvantage – low selectivity – which results in frequent false detections.

There is a widespread notion that the measurement of low concentrations of combustible gases and their vapors with catalytic sensors is not acceptable due to the high level of thermo and electrical noise comparable with the response signal and a number of uncontrollable parameters, such as humidity (see Fig. 1).

Fig. 1 shows the response of catalytic sensors embedded in Wheatstone circuit. The experiments have been conducted in a chamber which emulated different conditions: wet atmosphere, dry atmosphere with and without 100 ppm and 1% of methane. We note that methane in this experiment, as well as in the experiments presented later in Fig. 3 and Fig. 4, is diluted by dry air. Fig. 1b shows that the response signal ΔV_1 in atmospheric air (humidity = 79%) is approximately 1.5 mV, which is much higher than the response signal ΔV_2 at 100 ppm of CH_4. This analysis demonstrates that the measurement approach based on the Wheatstone circuit cannot control the humidity influence. The sharp peak near the '0' level is associated with the circuit and the graphical transient processes from zero value towards the value to be measured.

To overcome this problem, we propose the modified Wheatstone circuit and a gas diffusion control mechanism described in the next sections.

3. Approach and results

3.1. Sensing circuit

In this work, we use the commercial catalytic sensors DTK-3 by NTC-IGD for experimentation. The sensor is of 9.5 mm height and 9 mm in diameter, its power consumption is 75 mW in the continuous measurement mode. The relatively low power consumption is achieved by applying a heater implemented as a 10 μm platinum micro wire in glass insulation (2 μm). The active sensor has a platinum micro wire covered by the porous gamma alumina oxide material which is used as a catalyst support for catalytically active

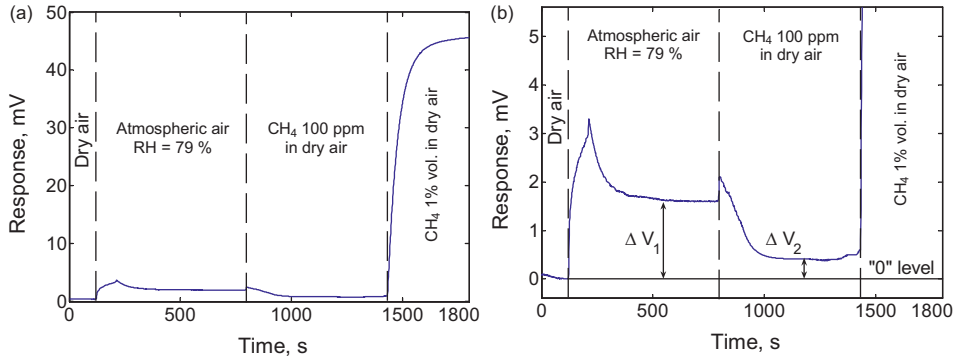

Fig. 1. The response of catalytic sensors embedded in Wheatstone circuit in the presence of various calibration gases and at continuous heating of the sensors: (a) response overview and (b) zoomed view.

Table 1
DTK-3 sensor parameters.

Parameter	Value
Supply voltage (Wheatstone sensing circuit)	2.8 V
Heating current	50 mA
Max. power consumption of one sensing element (SE)	75 mW
Resistance of one SE in normal conditions	10–14 Ohm
Effective range of methane concentration	0–5% vol.
Level of explosion isolation	Oexia*s*IIBT6
Output response setup time	<5 s

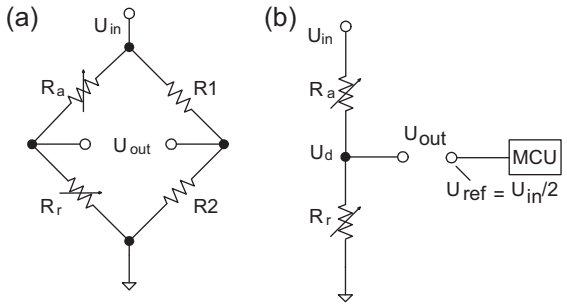

Fig. 2. (a) Wheatstone sensing circuit and (b) sensing circuit designed in this work where R_a and R_f are active and reference sensors, respectively, with a catalyst, but different sealed caps on top.

metals (mixture of Pd and Pt). In order to impregnate the catalyst support by the catalytic metal, salts of palladium chloride ($PdCl_2$) and platinum acid (H_2PtCl_6) are used. Table 1 summarizes the key parameters of the sensor.

The working principle of a catalytic sensor is the flameless burning of a combustible gas on the surface of a catalytically active substance. The heat generated by burning is proportional to the combustible gas concentration and changes the temperature of the sensor together with its resistance. Typically, the change in resistance is measured using the Wheatstone circuit shown in Fig. 2a. The circuit is supplied by voltage U_{in} and includes two resistors, R_1 and R_2, and two sensors, the active one R_a, and one for reference, R_r. The active sensor, which uses the catalytic material, is to perform the measurement. The reference sensor which is identical to the active one but not covered by the catalyst is therefore insensitive to the gas concentration in the atmosphere. The response of the sensing circuit is U_{out}, which shows the voltage difference between the two arms ($R_1 - R_2$ and $R_a - R_r$) of the sensing circuit. The reference voltage is generated by the arm $R_1 - R_2$.

For the detection of low and high gas concentrations we have modified the Wheatstone sensing circuit (see Fig. 2a). In particular, we keep active, R_a, and reference, R_r, sensors and exclude two resistors R_1 and R_2 to reduce the circuit power consumption. The fundamental difference from the classical Wheatstone sensing circuit is that we use two active sensors, i.e. two sensors with catalysts, but apply a diffusion limitation using sealed caps for the sensors. We discuss this approach in the next sections. The reference voltage, U_{ref}, in this case is set 'virtually', i.e. we define it as equal to $U_{in}/2$ by setting it at a 24-bit DAC MCU pin. Fig. 2b shows the sensing circuit used in this work.

3.2. Detection of low gas concentration

Our approach is based on diffusional squeezing of sensor response by introducing extra diffusional resistance to the reference sensor through the gas exchange sintered metal filter (up to 5–10% with respect to its actual value) [16]. In this case the reference sensor will have the parameters identical to the active sensor's ones including extra errors caused by non controllable components such as humidity, subtracted from each other in the Wheatstone circuit and, therefore, being compensated. Measuring the response has to be conducted after the third second when the response voltage is stabilized. However, the response amplitude in this case will be smaller than that of a standard catalytic sensor due to the signal subtraction from the residual burning in the reference sensor. In fact, the response signal's stability is much more important than its amplitude which can be amplified to the necessary level using hardware techniques.

The proposed approach can be realized by choosing active and reference sensors with most similar electrical and physical parameters. The parameters of interest are: current at supply voltage, sensor resistance in normal conditions, warm-up period, specific sensitivity to the gas to be detected. To ensure the diffusional squeezing of the reference sensor response, a sealed cap with a calibrated hole (0.1 mm in diameter) is put on the sensor. To guarantee the similarity of thermophysical conditions for both sensors in the presence of methane and moisture, the identical sealed cap with a calibrated hole 0.6 mm in diameter is put on the active sensor. With this sealed cap the diffusional squeezing of the response signal of the active sensor can attain as much as 50%. After the quick burning of methane in the reference sensor the measurement of response takes place. It is obvious that the hole diameter directly affects the time required for filling the volume of the sensor reaction chamber (33.5 mm^3) with a gas. We empirically defined that if the diameter is 0.5–0.6 mm this time is up to 10 s. In the case of the 0.1 mm diameter, the time is >100 s.

The transient response of both sensors has been measured during the measurement procedure including the heating of the sensors up to 450 °C in the presence of 1% CH_4 concentration (see Fig. 3). The transient processes finish within 5 s. These processes are associated with the inflow of water vapors into the chamber. The sharp peak appears (see Fig. 1b and Fig. 3) at the time of the

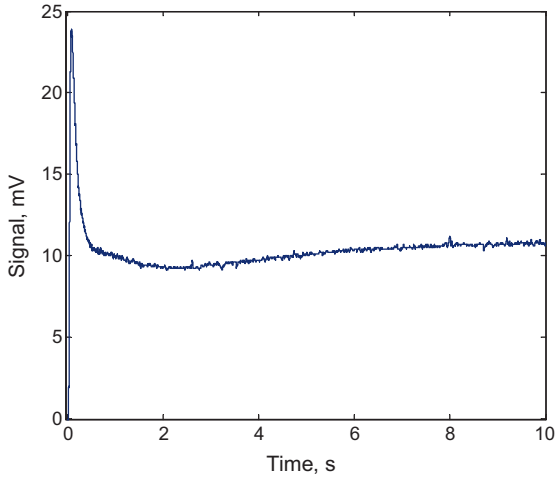

Fig. 3. Transient processes during the gas measurement procedure in the presence of 1% CH_4 (dynamic heating profile). The response signal is measured using modified Wheatstone circuit where the both sensors are covered with a catalyst and the active sensor has a sealed cap with 0.6 mm diameter hole and the reference one has a sealed cap with 0.1 mm diameter hole.

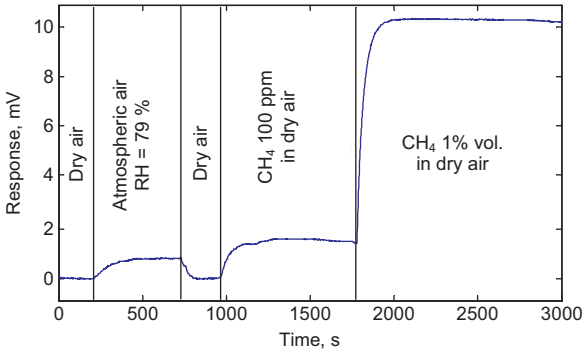

Fig. 4. Response of the sensors embedded in the modified Wheatstone circuit to various gases (continuous heating profile).

catalyst and water vapors reaction. This transient process is out of control. In this work, we do not study what happens with the catalyst during this process. The measurements are conducted after stabilization of the transient process–that is why the sharp peak does not affect the measurements.

The conducted experiment has shown that in order to guarantee the proper operation of the catalytic sensors embedded in the modified Wheatstone circuit and supporting the dynamic heating profile, the minimal heating period must be at least 5 s.

Fig. 4 duplicates the experiment shown in Fig. 1 and demonstrates the response of the modified Wheatstone circuit obtained following the proposed approach. It is evident that the sensor reacts on both humidity and gas. The gas signal must stand out above the background noise, i.e. humidity, which cannot be controlled. The advantage of our approach is that the influence of this 'noise' is excluded in the response to methane, as it is shown in Fig. 4 with respect to Fig. 1. It can be seen that the response to 100 ppm of methane (around 2 mV) is higher than response to humidity (around 1 mV), i.e. it is a reverse effect as compared to Fig. 1. We could, consequently, detect both 100 ppm and 1% of methane. We note, that in absolute numbers the response to CH_4 1% vol. in dry air in Fig. 4 is even less in absolute numbers than in Fig. 1, but the response to 100 ppm methane is higher than the response to humidity in Fig. 4. Unfortunately, the proposed solution can not entirely eliminate the influence of humidity due to the physical limitation of approximately 50 ppm in our case. Nevertheless, this

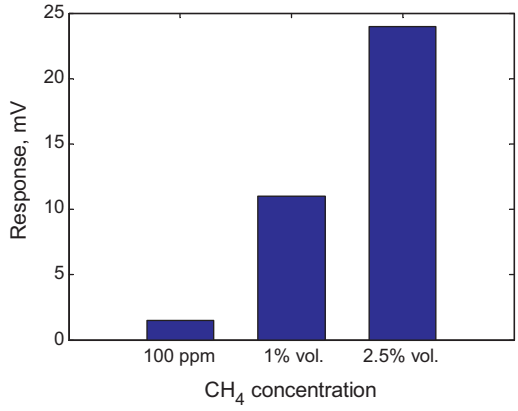

Fig. 5. Sensor circuit response in the presence of various methane concentrations (experimental results).

approach helps halve of the measurement error caused by the presence of a non controllable parameter, such as humidity. Thereby it ensures the catalytic sensor interoperability, i.e. improves the low threshold of the secure detection of gas up to 100 ppm.

Fig. 5 shows the sensor response in mV with respect to the hazardous gas concentration in the atmosphere.

3.3. Detection of high gas concentration

In a number of industrial applications there is an important requirement to catalytic sensors which is associated with their ability to monitor combustible and flammable gases, e.g. methane, with the increased concentration range starting below the LEL range and coming up to 100%. These applications are surely not related to living apartments or human presence spaces. However, it makes sense for dangerous gas monitoring in boiler facilities or gas mines [6] where a local methane concentration can be extremely high. The typical catalytic sensors are subject to carbonization—the effect when methane oxidation occurs without enough oxygen, which results in the formation of carbon-containing residue. Carbonization leads to the fall-off of the sensor sensitivity and even damage of the sensor.

To address this problem, we propose the sensing circuit composed of two catalytic sensors (active, R_a, and reference, R_r, ones) which are embedded in the modified Wheatstone circuit shown in Fig. 2b. In this case the reference sensor (R_r) is completely isolated from the atmosphere, the active sensor (R_a) remains with the same cap and the modified Wheatstone circuit works as the conductometric pair of the active (R_a) and reference (R_r) sensors. In the case of the gas absence, the heat dissipation on both sensors is equal, i.e. the sensing circuit is in the state of equilibrium. Methane does not react with oxygen and the thermal conductivity of methane is higher than that of air. Consequently, the presence of methane in the chamber of the active sensor results in the temperature decrease of its heating element which consequently reduces the active sensor resistance. This process changes the voltage U_d which shows the difference in the sensors' response. Also, U_d is the conductometric signal–the sensors' response which depends on the methane concentration in the atmosphere. From the viewpoint of the measurement procedure we define the circuit response U_{out} as follows:

$$U_{out} = U_{ref} - U_d \quad (1)$$

where U_{ref} is the reference voltage generated by MCU (see Section 3.1) and U_d is the sensors' response.

The objective of this measurement methodology is to alarm the monitoring system about a high methane concentration and

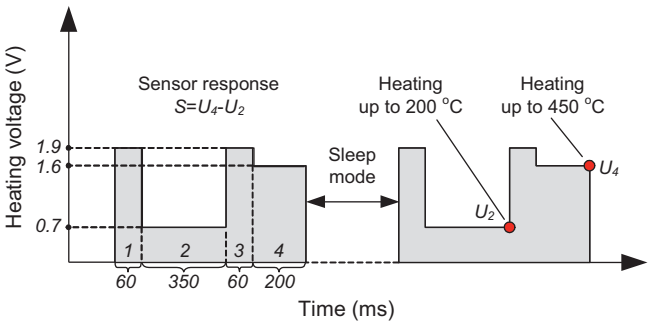

Fig. 6. Four stage dynamic heating profile.

turn off the measurement procedure before damaging the active catalytic sensor due to carbonization.

3.3.1. Measurement procedure

The proposed sensing circuit is similar to the one presented in [13] where the voltage divider circuit includes an active catalytic sensor and a resistor. In this work, we use a reference catalytic sensor instead of the resistor. The objective of this reference sensor is to guarantee the conductometric measurements of methane concentration. The conductometric measurement is carried out at 200 °C during the sensor heating using (1). At this temperature methane does not react with oxygen. If the methane concentration is less than 10%, vol., i.e. U_2 signal response is less than 5 mV (see Fig. 7), the measurement procedure continues to heat the sensor up to 450 °C and the methane concentration is defined [3] using (1). If the methane concentration is higher than 10%, vol., the measurement stops after the 2nd heating pulse and the system activates an alarm. When the concentration of methane is less than 10% vol., the sensing circuit keeps monitoring of the environment using the 4-stage heating profile and automatically excludes the influence of humidity on the measurements.

In Fig. 6 the dynamic heating profile is presented. There are four stages in the profile: two heating and two measuring ones. During the 1st stage which lasts for 60 ms at 1.9 V the sensor preheating is carried out. At the 2nd stage the moisture evaporation occurs without gas burning. By the end of this process which takes 350 ms at 0.7 V the sensor temperature is stabilized at 200 °C. It is when system performs 'reference measurement', or measuring of the sensor response U_2, thereby demonstrating how the measurement has been affected by the environmental conditions (without burning). This measurement is, in fact, analogous to the reference sensor's function in the Wheatstone sensing circuit [20]. The 3rd stage which takes 60 ms at 1.9 V ensures the heating of the sensor to a higher temperature when methane oxidation starts. Within the 4th stage lasting for 200 ms at 1.6 V the sensor operation is similar to that of the active sensor in the Wheatstone sensing circuit. By the termination of this final stage the temperature is stabilized at 450 °C and the system performs measurement of the sensor response U_4. The temperature of the sensing layer is in proportion to the environmental concentration of methane. Upon completing the measuring procedure, the sensor changes over to the sleep mode. Then this measurement cycle repeats.

If U_2 is higher than the preset value in the MCU memory in the presence of 10% of methane in the environment (U_2 signal is presented in Fig. 7), the 3rd and the 4th heating stages do not start. All the time when the high gas concentration is present in the atmosphere the sensor conducts the measurements using the first and the second pulses of the dynamic heating profile.

The output conductometric signal, C, of the catalytic sensor is well approximated by the empirical function (2).

$$C = K \cdot U^{1/3} \quad (2)$$

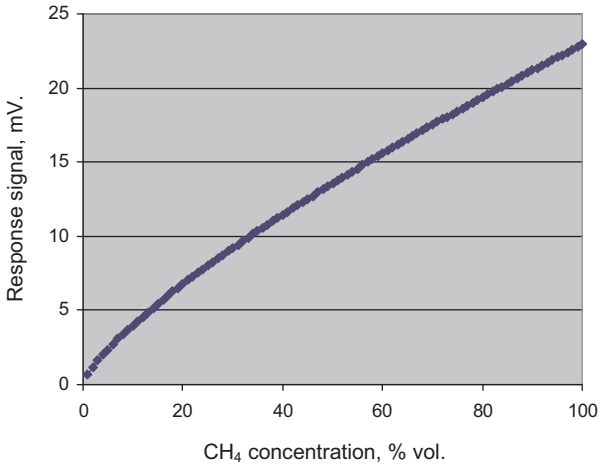

Fig. 7. Output signal of thermo conductometric sensor emulated by the active catalytic sensor at 200 °C.

where K is the factor characterizing physical properties of the sensing element and should be chosen during the sensor calibration, U is the sensor response and '1/3' is an approximate value.

3.3.2. Conductometric signal evaluation

It was empirically established that K in (2) is 1.6. The voltage divider with the active and reference sensors has a non-linear output conductometric signal in the range of 1–100% vol. methane (0.7 mV response in the presence of 1% vol. CH_4 and just 23 mV response in the presence of 100% CH_4), which is well approximated by eq. 1 with $K = 1.6$.

It should be noted that the output signal presented in Fig. 7 is the approximation of the real signal which reflects the temperature reduction and the sensor resistance due to the improved heat dissipation in the atmosphere in the presence of CH_4. The measurements were conducted at different methane concentrations in the dry air and at the room temperature. Experimental results demonstrate that 100 randomly chosen active-reference sensor pairs measure 90% vol. CH_4 and show the detected actual gas concentration in the range of 80–100%. The sensors were calibrated using (2) with $K = 1.6$. During the experiment all the sensors switched to "white scale". Switching was realized when the thermo conductometric signal U_1 had changed for 5 mV with respect to the calibrated value of 0 mV. The obtained results testify that the proposed approach helps securely detect, as well as prevent the carbonization of the sensor in the presence of high CH_4 concentrations in the atmosphere.

Fig. 7 shows that the sensor conductometric response is a non linear function of the gas concentration in the 0–100%vol. range. However, the catalytic response in the range of 0.1–10% vol. is linear. This feature has to be taken into account at the time of the sensor calibration.

Using the derived curve in Fig. 7 and the proposed four stage measurement procedure shown in Fig. 6, one can conduct the measurements of methane concentration in the 0.1–100% vol. range, given that the sensors operate in the catalytic mode within the 100 ppm–10% vol. range. If the 10% vol. threshold is exceeded, the sensor switches over to the conductometric mode of operation. In this case the sensor heating up to 450 °C is not required. This strategy helps prevent the carbonization of the catalyst of the active sensor and, therefore, degradation of the response in volts or even the sensor failure. If the measured value is less than the threshold one, the sensor is being heated and the measurement is conducted using the catalytic approach. The application of this measurement strategy is especially relevant in the case of sudden gas emissions, e.g. in a coal mine or in the event of a gas pipeline abruption.

4. Conclusion

In this work, we have demonstrated the approach for improving interoperability of the catalytic sensors, i.e. their ability of detecting various gas concentrations in the atmosphere, from 100 ppm up to 100% vol. instead of the widely applicable range of 0.1–10% vol. The increase in the detection range is enabled by the application of the proposed sensing circuit with two sensors embedded in a voltage divider circuit and the gas diffusion control mechanism. The advantage of this approach is the possibility of compensating non-controllable ambient conditions, such as humidity, during the measurement procedure.

The achieved results have got great potential for the industries dealing with the combustible and hazardous gases detection in indoor, outdoor or harsh environments. Our future work is aimed at the integration of this solution with the wireless sensor network paradigm.

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