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Miniature Gas Sensors Heads and Gas Sensing Devices for Environmental Working Conditions - A Review

Michal Borecki*, Mateusz Gęca, Mariusz Duk and Michael L. Korwin-Pawlowski

[1]Warsaw University of Technology

[2]Institute of Electron Technology

[3]Lublin University of Technology

[4]Université du Québec en Outaouais

***Corresponding author:** Michal Borecki, Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Poland; Tel: +48-22-234-7749; E mail: Borecki@imio.pw.edu.pl

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Abstract

There are many types of gas sensors. The paper presents the group of miniature gas sensor heads that may find applications in gas sensing devices oriented to work in environment conditions. These conditions in the near Earth surface are described as partial gases concentrations including highly varied concentrations of water vapor, atmosphere temperature and pressure as well as wind speed. The miniature gas sensors heads based on different sensing principles and systems of sensing devices are discussed.

Sensitivity and selectivity of heads and devices is discussed in the context of used technology. Nowadays, it seems that single miniature gas sensing head is characterized by too low sensitivity or to high cross-sensitivity to other gases and sensitivity to an environment parameters. Therefore sensing device should be prepared from set of miniature gas sensor heads, a gas sampling control module, as well as digital measurement data processing and control. The multiplication of heads in sensing device in homogenous set is used mainly for increase of sensitivity. While cross-sensitivity of sensing device is improved by hardware set of different type miniature gas heads or by introduction of thermal transducer and forced thermal cycle applied to single sensing element. Also the data processing in sensor device is essential for both cases of increase of sensitivity and decreasing of cross-sensitivity.

Keywords: Miniature gas sensors, Gas sensors heads, Gas sensing devices.

Introduction

The aim of this review is presentation of gas sensing devices as may be viewed from user [1] and constructor point of view [2,3]. A gas sensing element, gas sensor, gas sensor head and gas sensing device proper definition are somehow entangled. In common understanding, a sensor is an electronic component, module, or subsystem whose purpose is to detect actions or changes in its environment and show the information to other electronics devices. Thus, a sensor is always used with other electronics, whether as simple as a light bulb or as complex as a computer. Minimum sensor configuration consists of sensing element and basic electronic interface. Sensing element is used to converting information from not electrical form (energy) to an electric form, so it acts as a transducer. Sensor head is a set of sensing element and basic electronic circuits positioned in protection casing. Sensing device is a complete device equipped with at least one sensor head and essential electrical and mechanical interfaces, but it may consist of auxiliary units aimed for data processing, sensing complementary parameters and enabling digital communication. Moreover, technology readiness levels (TRL) of components reported as transducers, sensors, sensor heads and sensing devices may differ, see Table 1. As a result of such classification, example of methane sensing device at TRL7 is presented in Figure 1.

On the other hand of such classification, there are many types of advanced gas sensors. Most of them are costly devices the working principle of which enables precise gas concentration measurement in stable conditions, as for example in the laboratory set up presented in Figure 2.

The laboratory set-ups are often equipped with master gas sources that provide dry air and the examined chemical analytes [4]. The most significant disadvantage for environmental sensing

Table 1: Assumed technology readiness level of sensing device and its components, according to U.S. Department of Defense (DoD) definitions

Component, module, subsystem	Minimum technology readiness levels	Expected technology readiness levels
Transducer (sensing element)	basic principles observed and reported (TRL1)	proven through successful mission operations (TRL9)
Sensor	basic principles observed and reported (TRL1)	component validation in relevant environment (TRL5)
Sensor head	component validation in laboratory environment (TRL4)	system prototype demonstration in an operational environment. (TRL7)
Sensing device	subsystem model or prototype demonstration in a relevant environment (TRL6)	actual system proven through successful mission operations (TRL9)

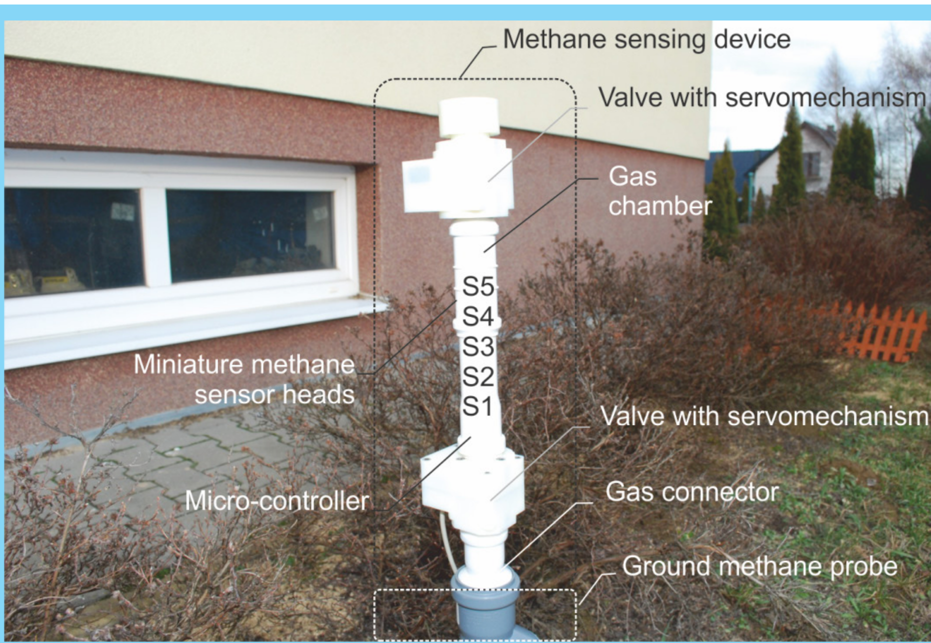

Figure 1: Example of methane sensing device, aimed to work on soil probe, realized at TRL7, during test in real environment

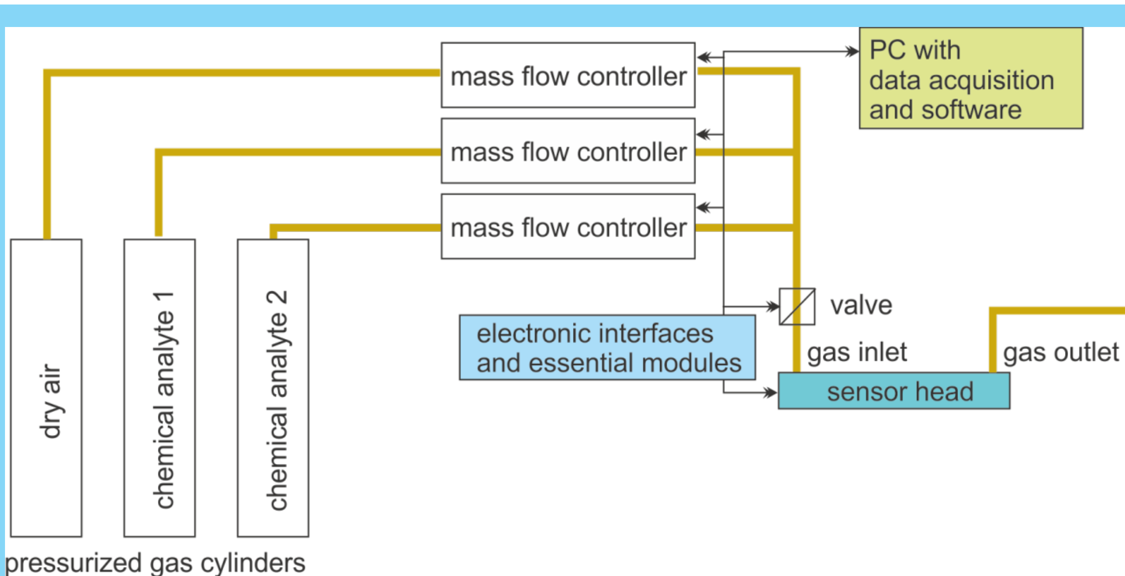

Figure 2: Example of scientific laboratory set-up for examination of gas sensors

of such set-ups is the lack of consideration of water gas presence in air, which in the Earth's air is one of the basic components.

Classical gas sensors include heads equipped with gas chambers where gas concentration transducers and additional sensors are positioned, electronic circuits for signal amplification and conversion, some essential modules for physical signal conditioning, and gas valves, gas pumps that together reproduce closely in the sensor construction the laboratory set-up [5].

The additional sensors are oriented to measure pressure and temperature. The sensor head may be relatively small [6], but the electronic interfaces and essential modules are often big and costly. For example, single mode tunable laser modules are sensitive to electrical discharge and require advanced electronic controllers of current, temperature and the optical power of a laser diode [7].

There is a lot of miniature gas sensing structures [8]. These structures may be used in miniature gas sensing heads, which can be used as components of gas sensing devices. Those devices are mostly used in practical applications outside the laboratory, in an environment. Therefore, the construction of miniature gas sensing structures and sensing devices has to be well thought-out for the particular environment conditions, device sensing parameters, costs of measurements and devices [9]. Moreover, the miniature gas sensing devices have to be as small as possible; therefore the contact of the gas and the transducer occurs mostly in the diffusion mode. This way the gas pump can be omitted in sensing device construction, but the gas sampling parts have to be carefully designed. It has to be noticed that environment gas measurement include selected gas concentration in an atmosphere that is a mixture of different gases described by their partial variable concentrations [10] as well as temperature and pressure, but also by the movement of the gas atmosphere [11]. In the terrestrial conditions the wind speed daily changes from 1 km/h to 20 km/h are quite common and the relative humidity changes range from 50 to 92 %RH, thus water condensation conditions at a temperature of 12°C are nothing unusual. Therefore, miniature sensing devices are often equipped with micro-heaters with the purpose of preventing water condensation at the gas transducer. In some cases the micro-heaters are an essential component of the gas transducer that enables the measurement [12] or increases the measurement speed [13].

The analysis of gas sensing devices may be performed from the sensing device point of view. There are several indicators that allow evaluating the performance of a gas sensing device: sensitivity, selectivity, detection limit, response time and recovery time. The basic gas sensing device components are the head and the electronic processing units. Most gas sensor heads are built from a few components, such as a selected gas parameter to electric signal transducer, electronic circuits oriented for direct information conditioning from the transducer, and complementary sensors, as presented in Figure 3. For the needs of this review the miniature gas sensor head dimensions are limited to not exceed 3 cm in all dimensions - width (W), length (L) and height (H).

The difference between the sensor head and the sensing device is the place of the required gas parameters analysis and presentation. In the gas sensing device there is the possibility of using multiple sensor heads, and of using additional components for gas sampling [14], or a neutral gas supply for head detoxification. The example of gas sensing device scheme is presented in Figure 4.

In the proposed scheme of a gas sensing device the gas sampling is performed with the use of micro-controller with a program dedicated to valve and micro-fan control. The gas sample procedure may include two sub-procedures: one for the examination of outside air temperature and its humidity, and the subsequent second mechanical sub-procedure which would include: valves opening, micro-fan activating, and waiting, micro-fan deactivating and closing valves. When the valves are closed the gas measurement can be performed.

In the proposed scheme of sensing device the valves may be omitted, but then the micro-fan has to be converted into an air flow sensor [15], and the protection of electronic circuits against condensation of water has to be designed and implemented [16].

Classical gas sensor classifications

Beyond sensors classification made from user point of view there are several scientific classifications of gas sensors [17]. The basic

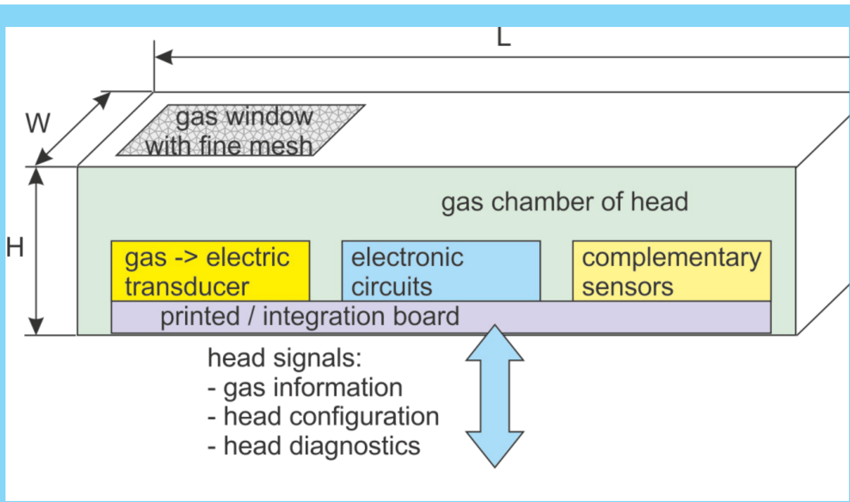

Figure 3: Miniature gas sensor head; {L, W, H} < {3, 3, 3} cm

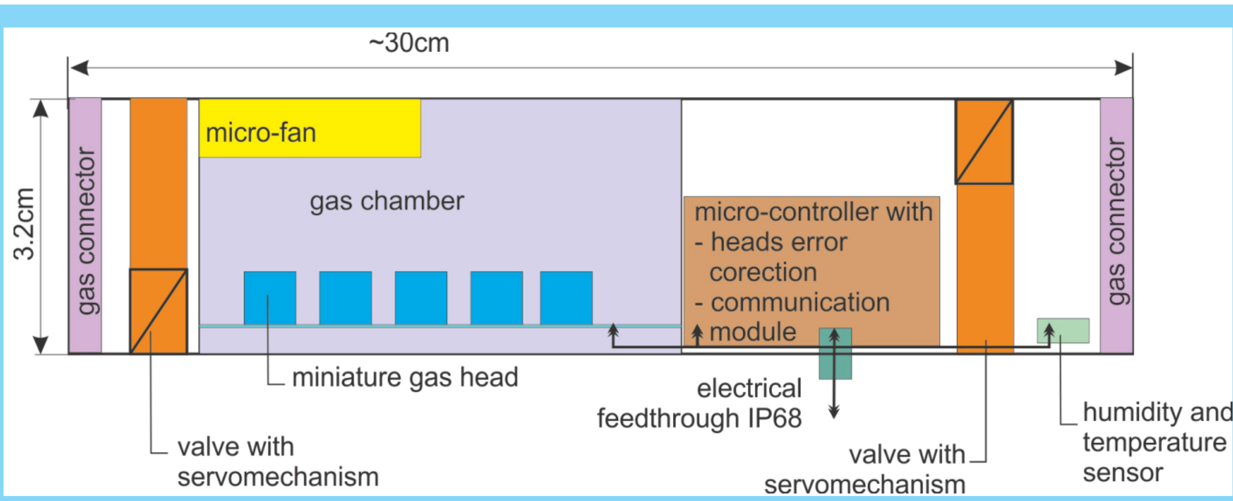

Figure 4: Example scheme of gas sensing device, prepared on base of [14]

classification is by the type of used sensing technology. This way the sensors are divided into a chemical and a physical group. The chemical sensors are based on chemical reactions while the physical sensors make use of physical phenomena. These groups of sensors are also called chemical reactive or physical reactive [18].

The physical gas sensors use ionization characteristics of gases, optical principles of specific gas properties, such as absorption or fluorescence or photo-acoustic phenomena [19]. The classical ionization gas sensors require maintaining a constant temperature and pressure of the characterized gas; therefore these type devices are not easy to miniaturize [20]. The optical gas sensor transducers include radiation sources, optical paths and radiation detectors. This type of devices can be relatively simple or may be very complex, with advanced optical path, and can be precise [21].

Chemical reactive sensors are also called electrochemical sensors. In this group the chemical reactions at the transducer result in most cases as a change of the element's resistance. The most popular reaction in electrochemical sensors is oxidation. For example, the oxidation and reduction equations of CO can be described as:

$$CO + H_2O \rightarrow CO_2 + 2H^+ + 2e^- \quad (1)$$

$$O_2 + 4H^+ + 2e^- \rightarrow 2H_2O \quad (2)$$

On the base of (1) and (2) the chemical sensors are sensitive to water and oxygen presence. However, due to need of reaction taking part, most electrochemical sensors are not suitable to measure concentrations of noble gases and gases that not easily reacts with oxygen, as for example CO_2. A micro-machined thin film electrochemical sensor that allowed for a simultaneous detection of CO_2, NO_2 and SO_2 has been created [22].

In some sensors both the physical and chemical phenomena are in use, as for example: in calorimetry, where the heat generated by a specific reaction is the source of analytical information [23], in micro-gravimetry where the relationship between the resonant frequency of an oscillating element and the mass deposited on its surface is the source of information [24].

The other gas sensor classification includes the number of transducers. Simple constructions are prepared with one transducer or channel. Two transducers enable the realization of sensor as a two-channel device or as device with an active reference signal. Sometimes in chemical sensors, the transducer power supply or its electric circuit connections are also used to perform the sensing function as the case of the amperometric, conductometric and chemical field effect transistors (ChemFET). In physical sensors such classification is not used, but transducers with a photodiode used in the reverse polarization regime or as photovoltaic source, as well as transducers using phototransistors as sensing devices are possible. Therefore, it seems that the essential classification of electrochemical sensors could be conveniently based on the type of the chemical material in combination with structure used in the transducer, as for example: organic polymer, metal oxide, and graphene, nanotubes of TiO_2 [25] or gold nanowires [26].

The gas sensor classification may base also on level of the development achieved, which may be defined as a proof of concept, laboratory model, prototype, or commercial. It should be noted that most scientific papers in the group of sensor journals present gas sensors at the proof of concept or laboratory model level.

Miniature gas sensors heads

The most typical miniature gas sensor heads include metal oxide, non-dispersive infrared (NDIR) and catalytic transducers.

Metal oxide gas sensor heads

Metals oxides are one of the most popular sensing materials providing high sensitivity and low cost of devices (MOS - metal oxide sensor, MOx sensors). The sensing technology usually utilizes transition metal oxides (e.g. WO_3, TiO_2) and posttransition metal oxides (SnO_2, ZnO), as they are characterized by more than one oxidation state, as opposed to non-transition metal oxides [27]. These materials from the electronic point of view are semiconductors. Due to their high conductance, n-type semiconductors are suitable for detecting reducing gases, while p-type semiconductors are usually used to detect oxidizing gases. SnO_2, ZnO, WO_3 are the most popular oxides used in gas sensing technology in hydrogen, oxygen or carbon monoxide measurements. An example scheme of metal oxide miniature head is presented in Figure 5a.

The phenomenon of the change of the resistance of heated metal oxides in the presence of the gas atmosphere is used in such sensors. The sensors may demonstrate significant cross-sensitivity, as a chemical reaction of oxidation depends on the composition of the gas atmosphere. Ambient humidity can also influence the conductivity of metal oxide as the water vapor adsorbed onto the sensing layer can act as a reducing gas, modifying the sensor response to oxidizing gases [28]. For example, sensors using SnO_2 are sensitive to a spectrum of combustible hydrocarbons and water [29].

The MOx sensor head principle of operation is based on heating the sensitive layer to the required temperature and measuring the change of the resistance of the sensing layer. The head sensitivity is usually defined as a ratio of R_{sa} to R_{sg} (or its reciprocal in case of oxidizing gases), where R_{sa} is the resistance of a sensor head R_s (see Figure 5b) measured with a reference gas, and R_{sg} is its resistance in a reference gas mixed with target gases. Sometimes the sensitivity can also be defined as the change in voltage (or current) in function of gas concentration depending on sensor head configuration in the sensing device. As an example, the simplest circuit of gas concentration sensing device based on voltage measuring on a load resistance is presented in Figure 5b). The detection of the gas concentration may be falsified by similar gases. An example of such situation is presented in Figure 5c). Therefore, optional gas filters are sometimes in use to absorb some interfering gases.

The operating temperatures of thin metal oxide-based sensors vary from 25°C to 500°C, which might cause potential selectivity problems [30]. The development in semiconductor technology allowed solving this problem by utilizing micro-heaters in the sensor's construction. MEMS and SiC micro-heaters can provide uniform temperature distribution, which is required to maintain optimal sensitivity and selectivity of the device [31].

Semiconductor metal oxide miniature gas sensors can successfully be used in indoor quality monitors and air cleaners. For example, Figaro's TGS 8100 provides high sensitivity to low concentrations of gas particles such as cigarette smoke or cooking odors, while TGS 5042 is characterized by sensitivity towards carbon monoxide providing detection range from 0 to 10000 ppm and output linear to CO concentration within its operating range.

Catalytic gas sensor heads

Catalytic combustion gas sensors have been wildly used in flammable gases and vapors detection for many years [32]. A combustible gas mixture can burn in temperatures lower than their ignition temperature in the presence of a catalyst. Reaction temperatures differ for different concentrations of examined gas. The principle of operation of catalytic gas sensors heads is based on the change of the resistance of the sensing element in the presence of exothermic oxidation of flammable gases (catalytic combustion) [33]. Catalytic gas sensors heads were classically based on coiled shaped platinum wires, sometimes coated with metal oxides. The planar structures for catalytic sensing are under development [34,35]. The technology progressed with the development of palletized resistor or pellistor. The pellistor consists of a very fine coil of platinum wire, embedded within a ceramic pellet, as presented in Figure 6a. On the surface of the

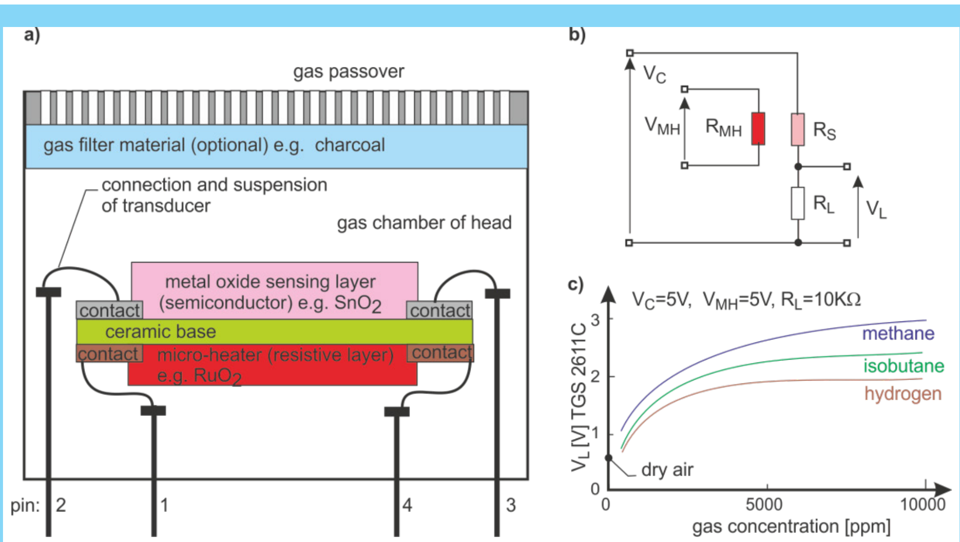

Figure 5: Example schematic of miniature metal oxide gas sensor head; a) schematic diagram, b) electronic circuit, c) sensitivity of TGS 2611C

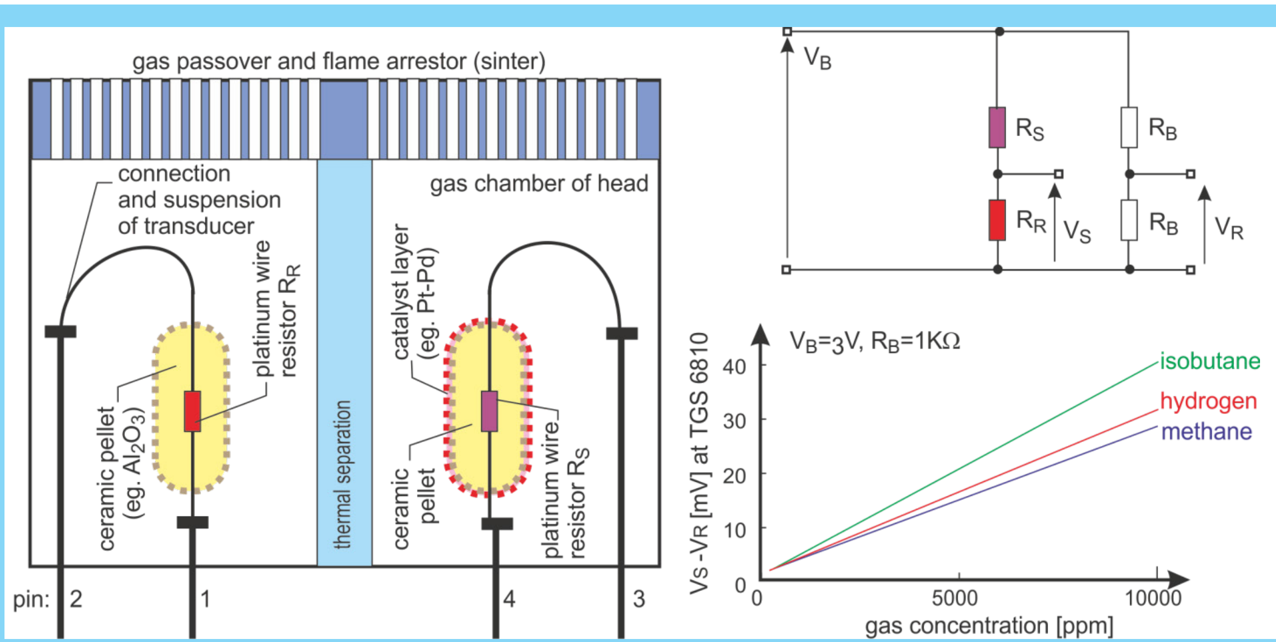

Figure 6: Example schematic of miniature catalytic gas sensor head; a) schematic diagram, b) electronic circuit, c) sensitivity of TGS 6810

pellet is a layer of a large surface area noble metal, which, when hot, acts as a catalyst. Pellistor sensors are always manufactured in pairs. The active sensing element of resistance R_S is supplied with an electrically matched reference element of resistance R_R which contains no catalyst layer (Figure 6b).

Working conditions of the reference element should ensure that no gas will oxidize on its surface. The Wheatstone bridge circuit is usually used as the readout circuit, allowing measuring the gas concentration. TGS 6810 is an example of a commercially available catalytic type miniature gas sensor head aimed for detection of combustible gases, as for example: methane, hydrogen, propane and iso-butane. As can be seen the differential signal amplitude from the catalytic heads, (see Figure 6), is relatively small, therefore an amplifying electronic circuits have to be positioned close to the transducers [36].

Nondispersive infrared (NDIR) sensor heads

Nondispersive infrared sensing is a simple spectroscopic method often used in gas examination. It is called nondispersive because the infrared beam in most cases passes through the head without deformation caused by the examined gas mixture. This is true as long as the mist is not present in the examined gas mixture. Unfortunately opposite conditions happen in Earth atmosphere quite often [37].

The main components of an NDIR sensor head are an infrared (IR) source, a gas chamber, a light filter and an infrared detector. There are many possible components and NDIR head configurations. The radiation source can be prepared as a bulb, a micro-heater, a light emitted diode (LED) [38] or a laser diode (LD). The radiation detector can be realized with a photodiode or a photoconductive element, for example made from lead sulfide (PbS) or lead selenide (PbSe). NDIR heads are often equipped for detection of a few optical wavelengths to minimize cross sensitivity of the head to environmental conditions and components drifts. Multi wavelength detection in miniature NDIR heads may be realized with one broadband source and a matrix of detectors equipped with optical filters, or may be realized with independent opto-pairs of source and detector.

The miniature NDIR heads dimensions, technology and costs limit the number of possible optical paths to one or two. To compensate for the drift of components in radiation sources with two optical paths, the configuration of the first path is used for gas sensing and the second path is used to create a reference signal. Complementary sensors can be used to detect the presence of the mist of water and temperature variations with concern for maintaining the objectives of miniaturization of the NDIR sensors, as is presented in Figure 7.

Micro-heaters are used in NDIR sensors to prevent variations in humidity and water vapors condensation inside the sensor's head unit. TDS 0034 is an example of commercially available NDIR type miniature gas sensor head that is aimed for detection of hydrocarbons as for example: methane, propane, butane and iso-butane [39].

Sensing devices for gas monitoring in environment conditions

The two main groups of sensing devices for gas monitoring in environment conditions may be oriented towards maintenance-free applications and hand – held instruments [40]. Both groups of instruments have to be described by sensitivity, selectivity, reliability and allowed working conditions. Basing on the analysis of miniature gas sensor heads, it is clear that consideration of all

Table 2: Cross-sensitivity of different miniature gas sensor heads

Type of gas sensor head	Example of head		Signal at 10 000ppm [V] (% of methane scale) of		
	head type	**head diameter/height [mm]**	**hydrogen**	**methane**	**isobutene**
Metal oxide	TGS 2611C	9.2/7.8	2.0 (66%)	3.0 (100%)	2.4 (80%)
Pellistor	TGS 6810	9.3/12.6	0.030 (107%)	0.028 (100%)	0.040 (143%)
NDIR	TDS 0034	20.0/16.6	0.400 (0%)	0.700 (100%)	0.850 (150%)

the mentioned parameters is difficult in sensing devices equipped with only one head. The mentioned previously miniature heads based on metal oxide, pellistor, and NDIR oriented for flammable and explosive gases detection are described by their range of output signal amplitudes and different type of cross-sensitivity, as presented in Table 2.

Despite this, sensing devices in the form of a sensing module with one sensing head and an electronic interface exist on the market. The electronic interface often consists of a signal amplifier equipped with a temperature compensator of head parameters realized with the use of a resistive sensor of temperature [41]. For example single head TGS 6810 is used in the FCM6812 pre-calibrated module for combustible gas detection. The mentioned module provides analog output voltage, in the range 0-4.5V, proportional to hydrogen gas concentration, in the range 0-15000ppm, but the module is sensitive also to other flammable gases, such as methane, ethane, etc. This module is equipped with three electrical connections: ground, supply and output signal.

The practical approach to adjust to different working conditions, results in two different types of sensing device constructions.

The first type of gas sensing devices construction is used to increase the sensitivity and prolong the maintenance free time of operation. In such case the miniature heads of the same type are used, and error correction algorithms are implemented in an integrated microprocessor. For example the error of concentration measurement was reduced by using signals from 4 heads from the set of 5. The average signal is calculated for 5 heads. The calculated distance between each head in the set and the average signal was used to select 4 heads from which data are used for subsequent averaging and then for presentation [42]. Therefore, the proposed configuration enables automatic elimination of a broken head from the active set. Such systems are suited for optical heads where sensitivity and zero gas concentration measurement stability are not the best, but cross-sensitivity is less of a problem than in metal oxide and catalytic gas sensor heads. The view of such system electronics based on TDS0034 NDIR sensor heads is presented in Figure 8.

The micro-controller unit in such device enables data preparation for presentation that includes averaging and exponential smoothing procedures. An example of results of

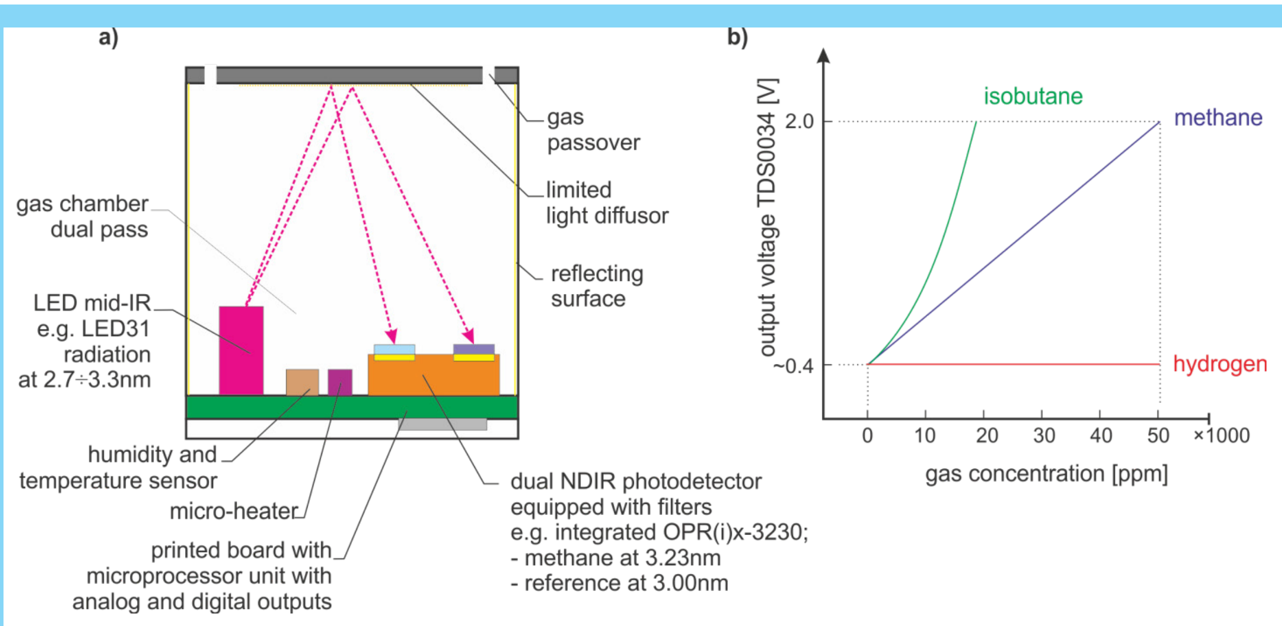

Figure 7: Example schematic of miniature NDIR gas sensor head; a) schematic diagram - reprinted from [14], b) sensitivity of TDS 0034 scaled for methane

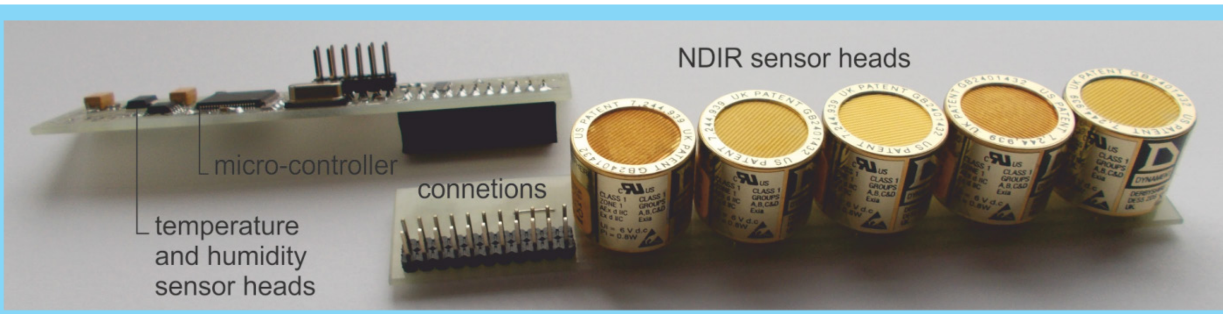

Figure 8: Multi-head sensing system with miniature gas sensing head of one type (NDIR)

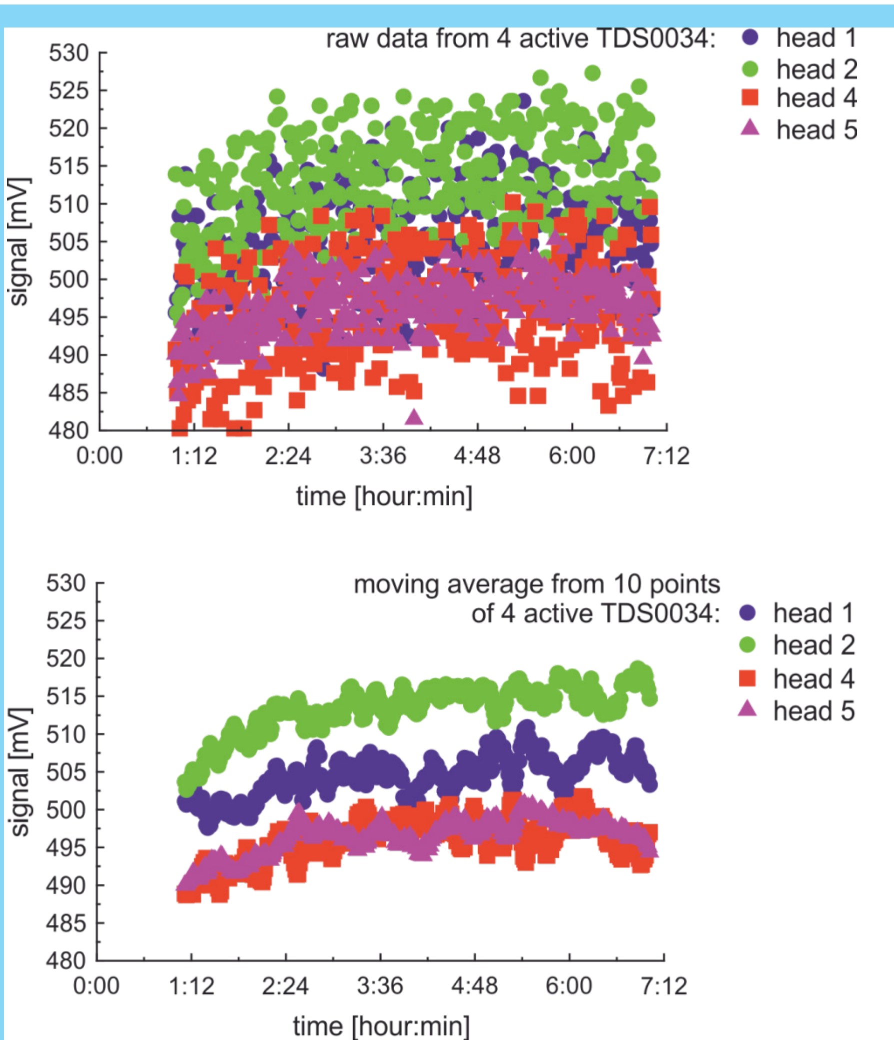

Figure 9: The sample results of raw data processing, with the use of moving average

raw data processing done using the principle of moving average from the head set of Figure 8 is presented in Figure 9. The data processing from miniature NDIR gas sensor heads seem to have significant influence on the presented results, as the output signal scale from 0.4V up to 2.0V is not wide compared to the range of measured gas concentrations and the noises of electronic raw signal converting and processing. The biggest disadvantage of such NDIR sensing devices is lack of selectivity between gases similar in structure, as for example CH_4 and C_2H_6 [43].

The construction of the second type of gas sensing devices has as objective the increase of their selectivity. It bases on different type of miniature heads of gas sensors. But connecting of different types of miniature heads require some dedicated conditioning circuits as the direct signals analysis performing may be difficult due to different signal levels [44]. Analysis is commonly realized in software after signal digitization. Therefore implementing nonlinear signal processing from a set of different miniature heads, as for example preparing dedicated artificial neural network is possible [45]. In such approximation the gas concentration can be measured more precisely than from one head device [46] or detection of gas composition becomes possible [47]. Moreover, the selectivity of gas type classification can reach 93% [48]. What is interesting, such performance may be even realized for miniature sensor heads using one technology but with transducers working in different conditions [49-50]. But using one head with forced thermal measurement cycle increase significantly time of gas analysis [51].

Proposition of gas sensing devices construction and technology are presented Table 3.

Summary

Gas sensing devices working in environment conditions have to measure partial gas concentration in highly variable gas mixtures. An important consideration in environment applications of gas sensing devices is the cross-sensitivity of miniature gas sensor heads to humidity, temperature and sometimes to oxygen presence in examined gas. The basic limitations of miniature gas sensor heads, according to type of transducer, are:

metal oxide sensors are sensitive to humidity, temperature, and oxygen presence in examined gas as depicted equation (1) and (2), also can be poisoned by some gases result in wrong reading of gas concentration,

catalytic sensors require the presence of oxygen to detect gas, and work at elevated temperatures often higher than 500°C, which can exceed safe conditions for maintenance-free use,

non-dispersive infrared sensors are sensitive to mist and when operated with integrated bulb radiation sources they can break with the signal of high gas concentration.

The transducers of miniature gas sensor heads are also characterized by relatively high cross-sensitivity to other somehow similar gases presented in the sensing gas mixture, which causes low selectivity. But, different type of transducers are characterized by different type of cross-sensitivity enabling realization of gas sensing device with quite good sensitivity, selectivity and reliability, when gas sampling parameters are measured or defined. For this purpose the gas sensor matrix can be made from miniature heads, auxiliary sensors of humidity, temperature and gas flow, as well as micro-heaters and proper data processing. Progress in gas sensing devices is related to materials, technology of integration, and also to the signal conditioning and data processing.

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Authors' contribution: Michał Borecki designed CH4 sensing device analyzed data from heads and devices, Mateusz Gęca designed and performed tests of heads and sensing device components, Mariusz Duk developed technology of integration and realized sensing devices, Jan Szmidt performed investigation of thin layers of gas sensor materials, Michael L. Korwin-Pawlowski controlled data analysis and construction assumptions.

Table 3: Proposition of gas sensing devices construction and technology

Type of gas sensor head set	Example list of transducers	Solved problem	Sensing device technology	Example of propositions
Mixed set	metal oxide, pellistor, NDIR	cross-sensitivity	nonlinear data processing from heads with the use of artificial neural networks	Hajovsky R, Pies M. (2012); Benrekia F, Attari M, Bouhedda M. (2013); Varun O, Paramartha D, Hiranmay S, Sugato G. (2012); Godoy AJC, Pérez IG (2017)
Homogeneous set	NDIR with LED sources	low sensitivity	statistical data processing with error correction methods	Borecki M, Duk M, Kociubiński A, Korwin-Pawlowski ML, (2016)
Single head	metal oxide, micro-heater, temperature control of metal oxide	cross-sensitivity	introduction of forced thermal cycle of sensing element and data evaluation on the base of mathematical model	Albrecht T, Matz G, Hunte T, Hildemann J, (2002); Kato Y, Mukai T, (2007); Seifert R, Keller HB (2017)

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