The Detection Properties Comparison for the SnO$_2$ Gas Sensor in Different Heating Regimes and the Step Change in Concentration

Libor GAJDOSIK

Department of Telecommunications, Faculty of Electrotechnical Engineering and Computer Science,
VSB–Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava-Poruba, Czech Republic

libor.gajdosik@vsb.cz

Abstract. *Non-periodical heating regimes and also periodical heating regimes were applied for the sensors TGS 813 and SP 11. Ethanol vapour in air was used as a detected substance. It was found that the sensitivity is better in the periodical square-wave heating regime among the tested regimes. The stabilisation of the sensor conductivity, when the concentration abruptly varies its value, takes longer compared to the constant heating regime at maximum heating voltage.*

Keywords

Dynamic response, semiconductor gas sensor, temperature modulation, thermal cycling, tin dioxide.

1. Introduction

Tin dioxide gas sensors have been used as detectors of the occurrence of gaseous substances in the air in numerous applications for years. In each of these applications, the occurrence means the change of the concentration of a detected substance in the sensor surrounding. Sensors are often operated in the heating regime dependent on time, because of better detection properties compared the constant voltage heating regime [1], [2], [3], [4], [5], [6], [7], [8], [9], [10], [11] and [12]. The overview of the often used heating time-dependent regimes is presented in [17]. It follows, that detection properties of the sensor are affected by its thermal inertia. The comparison of the detection properties of the sensor in different heating regimes when concentration is changed has not been carried out yet and this is performed here.

2. Experiments

The sensor was tested in the glass chamber of the volume 2700 ml. In the tested chamber, the atmosphere of clean air of 31 % relative humidity was used either with or without the concentration of 100 ppm of ethanol vapour. The concentration of 100 ppm was formed by the injection of the certain volume of the saturated ethanol vapour into the tested chamber filled with clean air of 31 % relative humidity. The saturated ethanol vapour was taken above the liquid in the closed bottle at the ambient temperature. The Antoine equation was used for the calculation of the saturated vapour concentration. The sensors were inside the chamber, heated by the heating voltage and the responses of the sensors were sensed. The response of the sensor is a term used here to refer to the electrical conductivity. The heating voltage was realized by the following manners: the time independent constant value, the step change from one value to the next one, the non-periodical consecutively increasing linear function, denoted here the ramp function, and periodical functions. The periodical functions were formed by the following formulas and used in the programmable voltage source, which heated the sensors: the sine-wave function was formed according to Eq. (1) and Eq. (2):

$$U(t) = U_S + U_A \sin(\omega t), \tag{1}$$

$$U_S = \frac{U_M + U_m}{2}, \qquad U_A = \frac{U_M - U_m}{2}, \tag{2}$$

where t is the time, ω is the angular frequency, U_M is the maximum value of the heating voltage a U_m is the minimum value of the heating voltage, the periodical symmetrical square-wave function was formed according to Eq. (3) and Eq. (4),

$$U(t) = U_m \quad \text{for} \quad 0 \leq t < \frac{T}{2}, \tag{3}$$

$$U_t = U_M \quad \text{for} \quad \frac{T}{2} \le t \le T, \qquad (4)$$

and the symmetrical triangle-wave function according to Eq. (5), Eq. (6) and Eq. (7),

$$U(t) = U_S + \frac{4U_A}{T}t \quad \text{for} \quad 0 \le t < \frac{T}{4}, \qquad (5)$$

$$U(t) = U_S + 2U_A - \frac{4U_A}{T}t \quad \text{for} \quad \frac{T}{4} \le t < \frac{3}{4}T, \qquad (6)$$

$$U(t) = U_S - 4U_A + \frac{4U_A}{T}t \quad \text{for} \quad \frac{3T}{4} \le t < T. \qquad (7)$$

The values $U_M = 5$ V, $U_m = 2$ V, 2.5 V and 3 V were chosen respectively. The time for setting next value of the heating voltage was chosen considering practical experiences $t_s = 1$ s. The response was sensed just before setting the next value of the heating voltage. The sensor response was sensed in 32 equidistant points during one period T of the heating voltage. It leads to the frequency of the principal harmonic component of the heating voltage $f = 1/(32 \cdot t_s) = 0.0312$ Hz. The ramp function was formed according to Eq. (5). Its value was changed in time sufficiently slowly, the value of the response at the given value of the heating voltage can be considered independent of time. The concentration of ethanol vapour in the air was kept either constant equal 100 ppm or its step change in time either from 0 to 100 ppm or from 100 ppm to 0 was used. The step change of the concentration was carried out by the following manner. In the case of the step from 0 to 100 ppm, the certain volume of the ethanol vapour was injected into the gas chamber to form 100 ppm concentration while the sensors were inside. In the case of the step from 100 ppm to 0, the sensors were taken out of the chamber and immediately put into the second chamber filled with clean air of 31 % relative humidity.

3.   Theory

The sensor SP 11 is formed on the alumina substrate on which the measuring electrodes are printed and covered by a metal oxide detection layer. A heater is printed on the reverse side of the substrate [13].

The sensor TGS 813 consists of the heating system realized by a resistive wire coil, which is located inside an alumina ceramic tube. The surface of the ceramic tube is covered with a pair of electrodes positioned to measure the electrical conductivity of the detection metal oxide layer deposited on the electrodes [14].

A detected substance in gaseous surrounding of the sensor must first diffuse into the detection layer to be detected. The detection layer in both cases is a polycrystalline porous structure of small pellets which touch each other and the space between them is filled with the air. A chemical substance in the gas phase can be detected, if the substance diffuses into the space between the pellets.

The electrical voltage heats the resistive conductor and heat penetrates through the ceramic layer into the detection layer. The detection of the gas became after the specific temperature of the detection layer was reached, which was manifested by the change of the electrical conductivity of the detection layer. It is possible to simplify the solution for transient heat conduction in the sensitive element according the literature [15]. The alumina substrate in the case of SP 11 is a plate of 2×2 mm dimensions and of 0.3 mm thickness. The alumina substrate in the case of TGS 813 is a cylinder of the similar size - the wall of the thickness 0.3 mm and of the length 2 mm. In both cases heat penetrates through 0.3 mm thick layer. The thickness is considerably smaller than the length and we consider transient heat conduction for relative short time after an abrupt temperature change at the beginning. According the literature[15] it does not matter on the shape of the object and we can consider it as an infinite plate of the finite thickness. It follows, that the Fourier Kirchhoff equation can be solved in one dimension. The sensing element of the sensor is surrounded by quiet air. The heat transfer coefficient of the air can be estimated $h = 8$ W$\cdot$m$^{-2}\cdot$K^{-1} [15] and alumina thermal conductivity $k = 20$ W$\cdot$m$^{-1}\cdot$K^{-1} [16]. It follows that air acts as a good thermal insulation of alumina substrate and during the transient state practically no heat penetrates from the plate into the air. Thus, it is possible to do further simplification. The alumina substrate can be replaced by the infinite half-space $x \ge 0$. The plane $x = 0$ represents the site of the heating system. At first the system is at uniform temperature ϑ_0 for all x. In the time $t = 0$ the step change of the heating is carried out to the temperature ϑ_1.

Transient heat conduction in the sensor can be solved by the Fourier Kirchhoff equation. We consider heat conduction in the half-plane $x \le 0$:

$$a\frac{\partial^2 \vartheta}{\partial x^2} = \frac{\partial \vartheta}{\partial t}, \qquad (8)$$

where a is the thermal diffusivity of the material (m$^2\cdot$s^{-1}), ϑ is the temperature and t is the time. The heating system is supposed to be in $x = 0$. Let us assume that the condition is given by the following equation:

$$\vartheta(x, 0) = \vartheta_0, \qquad (9)$$

where ϑ_0 is the initial temperature at the time $t = 0$ equal in every point of x. In the time $t = 0$ the step change of the temperature is carried out in the line $x = 0$ to the temperature ϑ_1. Then it is true:

$$\vartheta(0, t) = \vartheta_1. \qquad (10)$$

We calculate the distribution of the temperature in arbitrary point $x \neq 0$ at the time t. The Laplace transform can be applied on both sides of Eq. (8). The real variable of time t is transformed into the complex variable p. Let us designate the Laplace transform of the temperature $\Theta(x, p)$. We obtain the Laplace transform of Eq. (8) in the following form:

$$p \, \Theta(x, p) - \vartheta_0 = a \frac{\mathrm{d}^2 \Theta(x, p)}{\mathrm{d}x^2}. \tag{11}$$

Equation (11) is an ordinary differential equation, because the derivative of $\Theta(x, p)$ with respect to x does not depend on p. The associated homogeneous equation is:

$$\frac{\mathrm{d}^2 \Theta(x, p)}{\mathrm{d}x^2} - \frac{p}{a}\Theta(x, p) = 0. \tag{12}$$

The characteristic equation of Eq. (12) is:

$$\lambda^2 - \frac{p}{a} = 0. \tag{13}$$

The roots of Eq. (13) are:

$$\lambda_{1/2} = \pm \sqrt{\frac{p}{a}}. \tag{14}$$

The general solution of Eq. (12) is in the form:

$$\Theta(x, p) = K_1(p) \, \mathrm{e}^{\lambda_1 x} + K_2(p) \, \mathrm{e}^{\lambda_2 x}, \tag{15}$$

where $K_1(p)$ and $K_2(p)$ are proper coefficients, which are independent of x but they are dependent on p. The temperature must be finite $\Theta(x, p) < \infty$ at $x \rightarrow \infty$, because in the half-plane $x \leq 0$ there are no additional sources of heat. This is why the complementary solution of the homogeneous equation is considered only in the following form containing the negative root λ_2:

$$\Theta(x, p) = K_2(p) \, \mathrm{e}^{-\sqrt{\frac{p}{a}}x}. \tag{16}$$

The particular solution of non-homogeneous equation Eq. (11) can be found by the undetermined coefficient method. The particular solution describes the steady state which is independent on x. It can be supposed a constant designated $\Theta(x, p) = B$. After substitution into Eq. (11) we obtain:

$$pB - \vartheta_0 = a\frac{\mathrm{d}^2 B}{\mathrm{d}x^2} = 0. \tag{17}$$

As B is independent on x, its derivative with respect to x must be zero. Then it is true, according to Eq. (17), that the constant B equals:

$$B = \frac{\vartheta_0}{p}. \tag{18}$$

The summation of the Eq. (16) and Eq. (18) makes the general solution of Eq. (11):

$$\Theta(x, p) = K_2(p) \, \mathrm{e}^{-\sqrt{\frac{p}{a}}x} + \frac{\vartheta_0}{p}. \tag{19}$$

We determine the coefficient $K_2(p)$ in Eq. (19) using the Laplace transform of condition Eq. (10):

$$L \, \{\vartheta_1\} = \frac{\vartheta_1}{p}. \tag{20}$$

We substitute Eq. (20) into Eq. (19). It must be true for $x = 0$, that:

$$\frac{\vartheta_1}{p} = K_2(p) \, \cdot 1 + \frac{\vartheta_0}{p}. \tag{21}$$

It follows from Eq. (21), that the coefficient $K_2(p)$ equals:

$$K_2(p) = \frac{\vartheta_1}{p} - \frac{\vartheta_0}{p}. \tag{22}$$

Then the solution of Eq. (19) has the following form:

$$\Theta(x, p) = \left(\frac{\vartheta_1}{p} - \frac{\vartheta_0}{p}\right) \mathrm{e}^{-\sqrt{\frac{p}{a}}x} + \frac{\vartheta_0}{p}. \tag{23}$$

The solution must be re-transformed into the time domain by using the inverse Laplace transform. According to [18], if:

$$L^{-1} \left\{ \frac{\mathrm{e}^{-\alpha\sqrt{p}}}{p} \right\} = 1 - \frac{2}{\sqrt{\pi}} \int_0^{-\frac{\alpha}{2\sqrt{t}}} \mathrm{e}^u \cdot \mathrm{d}u =$$

$$= \mathrm{erfc}\left(\frac{\alpha}{2\sqrt{t}}\right), \tag{24}$$

where α represents:

$$\alpha = \frac{x}{\sqrt{a}}, \tag{25}$$

and if:

$$\mathrm{erfc}\left(\frac{\alpha}{2\sqrt{t}}\right) = 1 - \mathrm{erf}\left(\frac{\alpha}{2\sqrt{t}}\right), \tag{26}$$

then it is possible to express Eq. (23) by using Eq. (24) and Eq. (26) in the following form:

$$\vartheta(x, t) = \vartheta_1 \, \mathrm{erfc}\,(z) + \vartheta_0 \, \mathrm{erf}\,(z),$$

$$\text{where } z = \frac{x}{2\sqrt{at}}. \tag{27}$$

If $\vartheta_0 < \vartheta_1$, then Eq. (27) describes the increase of the temperature. For example $\vartheta_0 = 150$ and $\vartheta_1 = 350$. If $\vartheta_0 > \vartheta_1$, then Eq. (27) describes the decrease of the temperature. For example $\vartheta_0 = 350$ and $\vartheta_1 = 150$. If we consider the temperature range between $(150 - 350^{\mathrm{o}}\mathrm{C})$, the thickness $x = 0.3$ mm, the thermal diffusivity $a = 1.2 \cdot 10^{-5}$ mm^2·s^{-1} (alumina [16]), the temperature reaches 90% of the target temperature approximately in 0.08 seconds.

4.   Results

4.1.   Test 1.

The sensor heating was maintained at the constant heating voltage U. The sensor response was sensed after the step change of the concentration from zero to 100 ppm of ethanol vapour in the air was carried out. The obtained results were similar for both used sensors. The results for the sensor TGS813 are presented in Fig. 1.
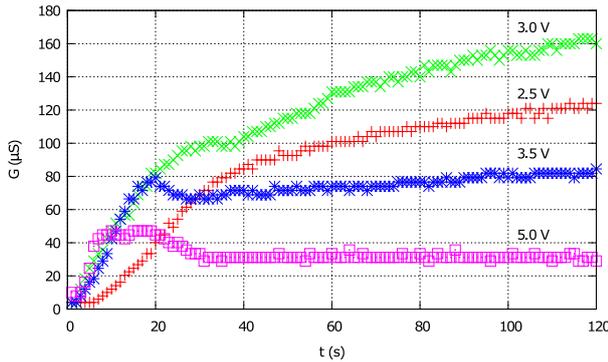
Fig. 1: The electrical conductance G of the sensor TGS813 when the step change of the concentration from 0 to 100 ppm of ethanol vapour was carried out. The constant heating voltage U is a parameter of the curves.

The experiment was repeated for different values of the heating voltage U. It is evident, that the response is dependent on the value of the heating voltage. The response reaches the greatest value for $U = 3$ V, but reaching the steady state value takes longer compared to the case for the voltage $U = 5$ V. The peak (with the duration approximately 25 s) appears in the responses for the voltages $U > 3.5$ V. The system of consecutive reactions between the detected compound and oxygen adsorbed in various forms [20] and [21] occurs to form reaction products during the detection. These phenomenons are dependent on temperature. The peak can be caused by a type of chemical reactions which occur at higher temperatures. It emerges from the experimental results, that it is proper to use higher heating voltage $U = 5$ V to obtain faster response despite the response reaches lower values here compared to the response for the heating voltage $U = 3$ V.

4.2.   Test 2.

The sensor was tested in the atmosphere of 100 ppm of ethanol vapour in the air. The heating voltage was kept at $U = 2$ V. The step change of the heating voltage was carried out from $U = 2$ V to $U = 5$ V and the sensor response started to be sensed. The value of 2 V was chosen experimentally, the sensor temperature is

too low to detect the substance, but high enough not to adsorb substances like water which can affect the detection process [21]. The value of 5 V was chosen considering the previous results, to reach quickly the steady state. Measured values of the response for the sensors TGS813 and SP11 are in Fig. 2.
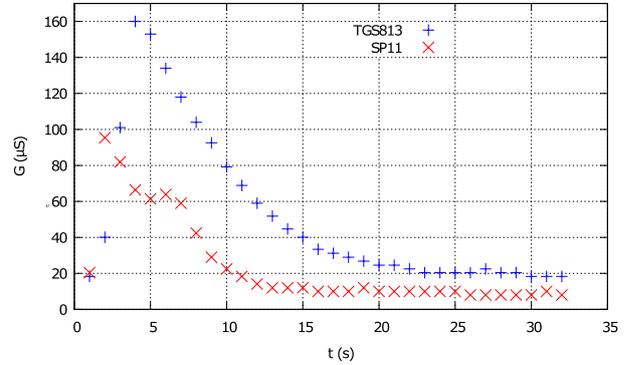
Fig. 2: The electrical conductance G of the sensors when the constant concentration of ethanol vapour in air and the step change of the heating voltage U from 2 V to 5 V were used.

4.3.   Test 3.

The sensor was kept in the atmosphere of 100 ppm of ethanol vapour in the air. The heating voltage was increased from $U = 2$ V to $U = 5$ V slowly by the step 20 mV per 10 seconds. The response was sensed 10 seconds after the change of the heating voltage and just before setting the next value of the heating voltage. The step 20 mV per 10 seconds was chosen experimentally, because the responses at given heating voltage are practically independent of time. Measured values of the response for the sensors TGS813 and SP11 are shown in Fig. 3.
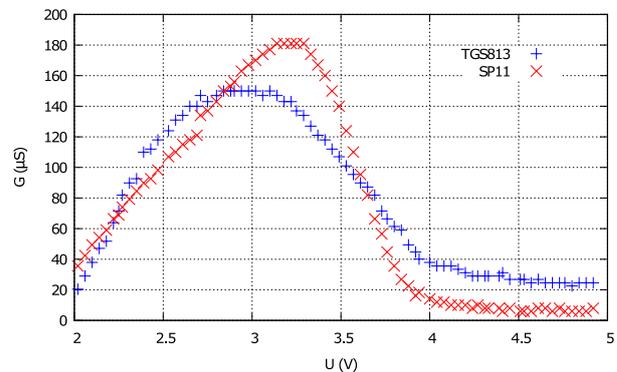
Fig. 3: The electrical conductance G of the sensors when ramp function for the heating voltage U and the constant concentration of the ethanol in the air were used.

It can be seen that the maximum lies in the range of heating voltage $2.5 < U < 3.5$ V of the electrical

conductance. This phenomenon can be used for explanation of the peak occurrence in the response in Fig. 2. When the step change of the heating voltage from 2 V to 5 V is considered, see Fig. 2, the temperature of the sensor quickly moves across the value associated with the maximum of the electrical conductivity in Fig. 3.

4.4. Test 4.

The sensors were tested in the periodical steady state heating regime. This means, that the sensors are heated by given periodical voltage in the tested chamber filled with the clean air until the response is periodically stable. It took approximately two minutes. An example of the response sensed in periodical steady state in the clean air is demonstrated in Fig. 4.
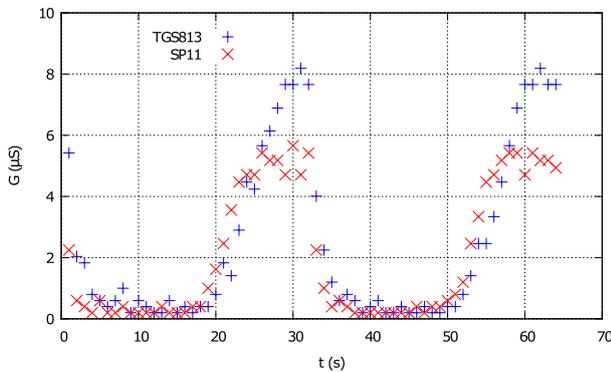
Fig. 4: The electrical conductivity G of the sensors when the periodical square-wave heating voltage of $U_m = 2$ V and $U_M = 5$ V and clean air were used. The record corresponds to two periods of the heating voltage.

At the beginning of the experiment, the concentration was abruptly changed from $0 - 100$ ppm and then the response started to be sensed. An example of such a response is in Fig. 5. The change of the concentration caused a transient state and the peaks of the response exceeded 120 µS. It is 2.4 times more then the peak of 50 µS in Fig. 1. This follows better sensitivity of the sensor in the periodical heating regime compared to the constant voltage heating regime.

When the periodical steady state of the response had been restored, see Fig. 6, the peak values of the response decreased to 55 µS. The peak values in Fig. 6 correspond to the peak values in Fig. 1, about 50 µS, where the constant heating voltage of 5 V was used. Then the experiment continued. The step change of the concentration was carried out from 100 ppm to 0 and the response was sensed. The time dependency of the response is exhibited in Fig. 7.

It follows, that the peak values of the response remain almost equal and decrease slowly in time.
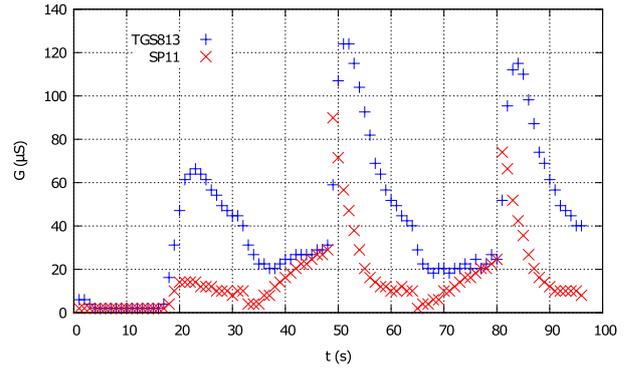
Fig. 5: The electrical conductivity G of the sensors when the periodical square-wave heating voltage of $U_m = 2$ V and $U_M = 5$ V was used. The step change of the concentration from $0 - 100$ ppm was carried out at the beginning of the record. The transient state caused by the step change occurs here. The record corresponds to three periods of the heating voltage.
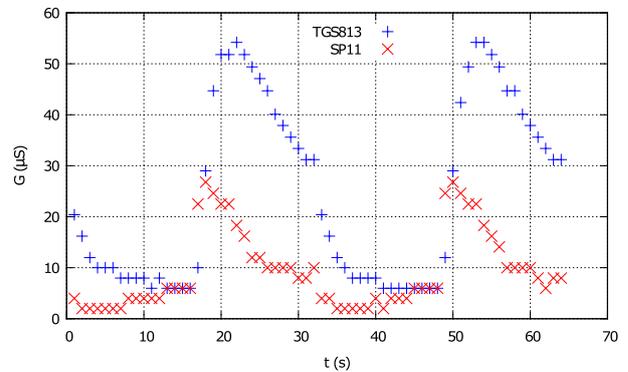
Fig. 6: The electrical conductivity G of the sensors when the square-wave heating voltage of $U_m = 2$ V and $U_M = 5$ V and the constant concentration 100 ppm were used. The record of the periodical steady state corresponds to two periods of the heating voltage.
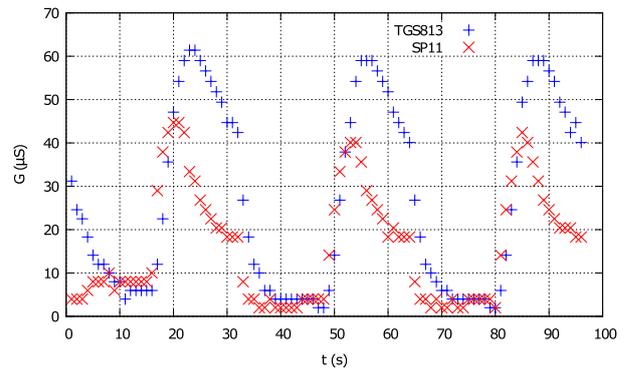
Fig. 7: The electrical conductivity G of the sensors when the periodical square-wave heating voltage of $U_m = 2$ V and $U_M = 5$ V and the step change of the concentration from 100 ppm $- 0$ were used. The transient state caused by the step change occurs here. The record corresponds to three periods of the heating voltage.

4.5.   Test 5.

The sensors were heated by the constant heating voltage $U = 5$ V in the atmosphere of 100 ppm ethanol vapour in the air until the time independent response was reached. Then the abrupt change of the concentration was carried out to zero and the responses of the sensors started being measured. An example of the responses is presented in Fig. 8.
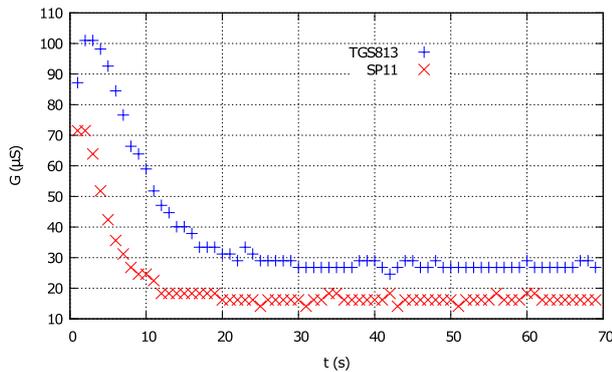
Fig. 8: The electrical conductivity G of the sensors when the constant heating voltage of $U = 5$ V and the step change of the concentration from 100 ppm – 0 were used.

It is demonstrated here, that the response reached its stability after 20 seconds or 10 seconds for TGS 813 or SP 11 respectively, which is considerably shorter time compared to the case in Fig. 7, where the response did not reach the value corresponding to zero concentration of the substance even after 100 seconds. It is possible to explain this phenomenon in the following way: the process of the consecutive chemical reactions, i.e. the rate constant and types of reaction products with different adsorption properties to the surface of SnO_2 [20] [22], [23] and [24], are affected by the variation of the temperature during a periodical heating regime.

4.6.   Test 6.

The sensors were tested in the steady state periodical heating regime. The voltage U_M was maintained at 5 V and the voltage U_m was maintained at values 2 V, 2.5 V and 3 V respectively. U_m was kept constant during given test. The concentration of ethanol vapour was used as the step change from 0 – 100 ppm, constant of 100 ppm and the step change from 100 ppm – 0 respectively for the measurement at given value of U_m.

Different range of heating voltages leads to different range of the temperature of the detection layer. It was found, that when the values of $U_m > 2$ V were used, the peak values of the responses associated to the step change of the concentration from 0 – 100 ppm are lower

compared to the responses at $U_m = 2$ V. The overview of the peak values is presented in Tab. 1.

Tab. 1: The peak values of the electrical conductivity G of the sensors. The square-wave heating voltage of $U_M = 5$ V and different values of U_m were used. The responses were sensed in the steady state periodical heating regime. G_r is the conductivity at the step change of the concentration from $0 - 100$ ppm, G_u is the conductivity at the constant concentration of 100 ppm, G_f is the conductivity at the step change of the concentration from 100 ppm $- 0$.

	G_r (µS)	G_u (µS)	G_f (µS)
$U_m = 2$ V			
TGS813	120	55	60
SP11	90	28	40
$U_m = 2.5$ V			
TGS813	65	70	50
SP11	45	60	35
$U_m = 3$ V			
TGS813	70	50	50
SP11	50	35	40

Identical experiments were carried out when the sine-wave and the triangle-wave heating voltage were applied to the heating system of the sensor. The same frequency ($f = 0.0321$ Hz) as in the square-wave case was used. The results are summarized in Tab. 2.

Tab. 2: Thepeak values of the electrical conductivity G of the sensors when different types of the heating voltage of the parameters $U_M = 5$ V and $U_m = 2$ V were used. The responses were sensed in the steady state periodical heating regime. G_r is the conductivity at the step change of the concentration from $0 - 100$ ppm, G_u is the conductivity at the constant concentration of 100 ppm, G_f is the conductivity at the step change of the concentration from 100 ppm $- 0$.

	G_r (µS)	G_u (µS)	G_f (µS)
triangle			
TGS813	90	50	160
SP11	45	30	100
sine			
TGS813	80	55	140
SP11	35	35	80

It follows from Tab. 2 and Tab. 1, that the peak values of the response are lower in the sine-wave case or in the triangle-wave case compared to the square-wave case when the range of the heating voltage of $U_m = 2$ V , $U_M = 5$ V is used. It can be concluded, that the square-wave heating regime is more advantageous, due to the higher sensitivity. The longer stabilisation time is the disadvantage of the tested heating regimes. The stabilisation time is a term used here to refer to the time, when the sensor response decreases to 110 % or increases to 90 % of the value of the response related to the steady state. But it is possible to modify the periodical heating regime by setting the constant voltage regime of $U = 5$ V after the detection of the occurrence of the substance until the response corresponds

to zero concentration. This could shorten the stabilisation time.

5. Conclusion

The experiments were carried out with the commercial sensors TGS813 and SP11. It was found, that the periodical square-wave heating regime is the most advantageous among the tested heating regimes. The stabilisation time of the sensor (time, when the concentration abruptly changes its value) is longer at the periodical heating regime compared to the constant heating regime at maximum heating voltage. It was suggested that a combination of both heating regimes, i.e. periodical and constant voltage, could shorten the stabilisation time and keep higher sensitivity of the sensor. Further research would be target to optimisation of the heating regime of the sensor.

Acknowledgment

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About Authors

Libor GAJDOSIK graduated in telecommunication engineering from the Czech Technical University (Prague, Czech Republic) in 1983. He received his Ph.D. in 1998 from the Technical University of Ostrava (Ostrava, Czech Republic). His thesis was focused on the detection properties of tin dioxide gas sensors at the dynamic operating modes. He has been an assistant professor at the Technical University of Ostrava since 1989. His main interests are electronics, circuit theory and chemical sensors.