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A Thin-Film Platform for Chemical Gas Sensors

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Abstract—In the study, a technique for formation of planar microheaters that make it possible to heat an active zone to a temperature higher than 500°C is proposed and successfully implemented. The developed heating elements are distinguished by low power consumption, short response time, and extremely high resistance to impact loads. The synthetic approaches used in the work (anodic oxidation, photolithography, and magnetron sputtering) feature manufacturability and scaling simplicity. This makes planar heating elements a promising platform based on which semiconductor and thermocatalytic sensors of toxic and explosive gases may be created.

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INTRODUCTION

Monitoring toxic (CO, NO_2, H_2S, SO_2, etc.) and inflammable (H_2, CH_4, and other hydrocarbons) components in the atmosphere is an immensely important issue for industrial safety. The niche of the application of gas sensors is tremendously broad, including enterprises in the oil, gas, and chemical industries, drilling units, mines, research and development laboratories, and residential homes. Stationary gas analyzers with wired power supply connections and remote sensors are gradually being substituted by wireless sensor networks that transfer information. This approach has a number of advantages:

(1) Quick and simple deployment of the system on a new object;

(2) Possibility to add an unlimited number of control points;

(3) Convenient mounting of remote sensors in hard-to-reach places;

(4) High failure tolerance level of the system on the whole.

Taking into account the scope and rate of the spread of wireless Internet, it is evident that the ubiquitous introduction of wireless networks will soon follow.

Note that, irrespective of the type of the employed gas control system and technique of taking readings from sensors, a sensing element (a primary transducer), which converts the concentration of the determined component in air into an electric signal, is always included.

In the current market of sensor systems, semiconductor and thermocatalytic sensors (sensing elements) have become the most widespread due to their simple construction, stable characteristics, and low cost price, with each one being applied in its own niche.

The content of inflammable gases in the air (e.g., methane, ethane, propane, and hydrogen) is controlled in order to rule out gas explosions. It is important to preclude exceeding the lower concentration limit of flame propagation (LCLFP), which is considerably higher than the limits prescribed by the sanitary code for toxic gases. For methane, which is the main component of natural gas, the LCLFP is 4.4 vol %. Most often, to tackle this issue, thermocatalytic sensors, also known as pellistors, are used. For analyzing the trace amounts of toxic substances in the atmosphere (at the level of parts per million), it is most practical to employ semiconductor sensors.

The principle of operating semiconductor gas sensors is based on the change in the conductance of a sensitive layer (which, as a rule, oxides transition metals, such as SnO_2, ZnO, and In_2O_3) due to the sorption of gas molecules on its surface [1]. This method can be used to detect gases with both the donor properties (e.g., various inflammable gases, including methane, propane, volatile organic compounds, CO, and NH_3) and the acceptor properties (ozone, nitrogen oxides, chlorine, fluorine, etc.). Note that to achieve a short (~1 s) response time of a semiconductor sensor, its sensing element needs to be heated to quite high temperatures (from 250°C for detecting ethyl alcohol and hydrogen to 500°C in the case of methane).

The principle of operation of thermocatalytic sensors is based on the flameless oxidation of methane (or any other inflammable gas) at the surface of a catalytically active element and measurement of the amount of heat released, which, under the constant conditions of the heat-and-mass exchange, is proportional to the methane concentration in air [2]. This principle of operation makes thermocatalytic sensors selective solely to inflammable gases. The catalytic oxidation of an inflammable gas proceeds rapidly only at high temperatures (in the vicinity of 400 to 500°C when using a Pt–Pd catalyst). Therefore, the presence of a heating element is a necessary condition for both semiconductor and thermocatalytic sensors. The drawbacks of commercially available pellistors are their high power consumption (higher than 100 mW) and low resistance to impact loads, which limits their application in portable devices for the analysis of the gas atmosphere.

In recent years, miniaturized planar sensing elements for gas sensors exhibiting extremely low power consumption and a high level of mechanical strength have been actively researched and developed [3]. They are produced by low-cost mass production microelectronic technologies.

Thermal and mechanical stability, resistance against vibrations and impacts, low heat conduction, and a high degree of adhesion of the sensitive layer and metal contacts are among the key requirements for the substrate material of planar sensing elements of gas sensors. In practice, as a base, silicon oxide/nitride wafers, polycor (Al_2O_3), or porous anodic alumina films are employed. The main disadvantages of silicon-based devices are their low levlel of adhesion of a platinum heater to the membrane material, the low stability level of silicon nitride at high temperatures, and their high cost price.

An alternative solution is to form planar gas sensors on porous anodic alumina substrates [4, 5]. Unlike the silicon technology, this provides a number of advantages [6]. In particular, the open porous structure with the possibility to vary its parameters over a wide range, high thermal stability up to 1000°C, greater mechanical strength than SiO_2 substrates with the same thickness, the possibility to precisely control the shape of a porous substrate with micrometer resolution, and the strong adhesion of the metal and catalytically active layers with no need for intermediate buffer layers are among the key requirements made on the substrate material of the planar sensing elements of gas sensors.

EXPERIMENTAL

In the present study, the construction of a miniaturized planar heating element for semiconductor and thermocatalytic gas sensors is proposed. Below, a description of the key steps to obtain it and the techniques for analyzing its functional characteristics are given.

As a base for the creation of microheaters, anodic alumina films are employed. This material was formed by anodizing aluminum foil at a constant voltage. At the surface of the resulting porous oxide film (with a pore diameter of 150 ± 10 nm and a distance between the neighboring pores of 260 ± 40 nm), an array of planar heating elements of platinum in the form of a two-dimensional spiral (meander) with a lateral size of ~150 × 150 μm^2 was formed by photolithography and magnetron sputtering (Fig. 1). At the final step, the heater's surface was covered with a protective Al_2O_3 insulator layer. It is worth noting that applying a photolithographic approach to obtaining microheaters makes it possible to form hundreds of identical elements in a single work cycle, ensuring a low cost price and a high degree of reproducibility of the production.

For attestation of the electrophysical characteristics of the obtained elements, a selection of microheaters was mounted on the current-carrying seals using a Kulicke&Soffa 4526 wedge bonder for ultrasonic welding.

An important characteristic of the heater's material is the temperature coefficient of resistance (TCR), which needs to be known in order to evaluate the temperature of the sensor's active area from the electrophysical parameters. Note that the TCR of thin films can differ dramatically from that of bulk material [7, 8], which leads to significant mistakes when using reference data in the calculations. It the present work, the TCR of the sputtered platinum was determined as follows. A heating element mounted on a current-carrying seal, was placed in a Binder heating chamber, and the temperature in it was increased stepwise from room temperature to 130°C. In the course of the experiment, the current flowing through the meander was measured at a constant potential difference of 10 mV. Note that the mentioned supply voltage value was low and did not lead to the heating of the meander; therefore, its temperature was determined solely by the ambient temperature.

The dependence that the resistance and temperature of the Pt meander has on applied voltage was determined from the cyclic voltammetry data. The response time of the planar heating element was determined by testing the devices in the course of the cyclic change of the operation mode. The heater was alternately fed with voltages corresponding to a temperature of ~100°C (standby mode) and ~400°C (operating mode). The pulse duration was 1 s and the rate of data acquisition was 1000 points per second.

The images of the heating element in the course of operation were obtained in a darkened room using a Nikon Eclipse 600pol optical microscope.

The impact resistance of the planar elements was tested by the action of periodic impacts on the circuit

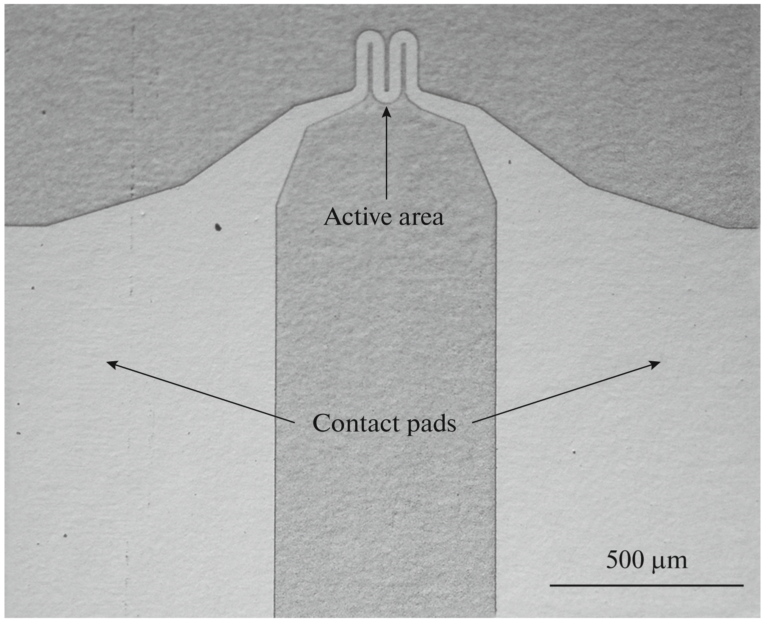

Fig. 1. General view of planar heating element. Active area in form of two-dimensional spiral and contact pads for mounting in electric circuit are shown. Image is obtained by optical microscopy.

board on which the current-carrying seal with a microheater was mounted.

RESULTS AND DISCUSSION

Temperature Coefficient of Resistance

In the general form, the dependence of the resistance on temperature is described as

$$R_T = R_0(1 + \alpha(T - T_0)). \quad (1)$$

Here R_T and R_0 are the values of the electric resistance at temperatures T and T_0, correspondingly, and α is the temperature coefficient of resistance (TCR), measured in inverse Kelvin. In establishing the TCR of the metals, T_0 is commonly assumed to be 0°C; therefore, R_0 is the resistance of the studied conductor at this temperature.

The resistance of a planar heating element rises when the temperature is increased due to electron scattering on the phonons (thermal vibrations of the lattice). To choose the parameters of operation of a device properly, it is necessary to know the exact TCR value. It is worth noting that the value α depends on the microstructure of the heater's material and the prehistory of obtaining it; therefore, using a reference value may result in a significant inaccuracy.

In the course of the experiments on determining the TCR, the temperature of the heating chamber in which the microheaters were placed was increased stepwise, which led to a stepwise change in the current of the circuit under a constant supply voltage. A typical view of the obtained $I(t)$ dependence is shown in Fig. 2a. Next, at each step, the electric resistance of the meander was determined at the corresponding temperature. The resulting dependence $R(T)$ is described well with a linear equation (Fig. 2b), which confirms that the TCR is constant at least in the employed temperature range. This allows us to determine α as a ratio of the slope to the free term:

$$\alpha = \frac{R_T - R_0}{R_0(T - T_0)} = \frac{\Delta R}{R_0 \Delta T} = \frac{1}{R_0}\frac{\partial R}{\partial T}. \quad (2)$$

The average TCR value over a series of 25 microheaters was $(34.3 \pm 0.7) \times 10^{-4}$ 1/K.

According to the literature [8], the TCR of noble metals and their alloys remains constant up to high temperatures; therefore, the values obtained as described above may be confidently used to compute the temperature of the heating element in a wide range of supply voltages. The experimental determination of the TCR value in the whole temperature range concerned (up to 500 to 600°C) is hardly possible because of the limited thermal stability of the materials of the components in the electric circuit (cover of the connectors and wire insulation).

Parameters of Operation of a Heating Element

The parameters of operation of a planar heating element (supply voltage and power consumption) are determined based on the cyclic voltammetry. The dependence of the current (I) on the applied voltage (U) recorded in the course of the linear sweep of the supply voltage at a rate of 50 mV/s is illustrated in

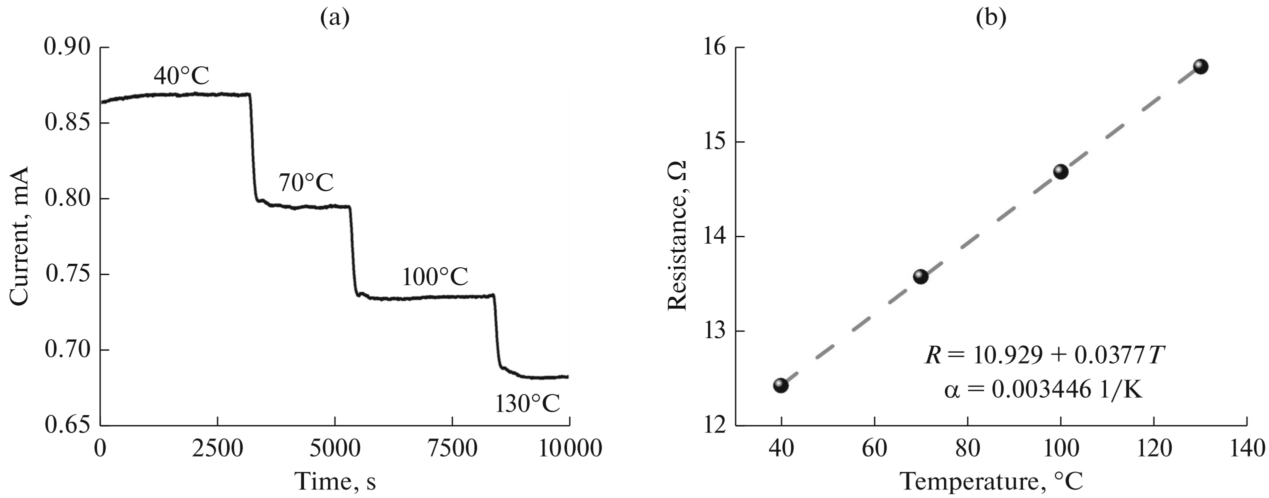

Fig. 2. Determination of temperature coefficient of resistance (α): (a) typical view of time dependence of current, recorded in course of experiment and (b) dependence of resistance (R) of planar heating element on temperature (T).

Fig. 3a. It is clearly seen that the behavior of the current–voltage characteristic (I–V characteristic) of the planar heaters deviates from a straight line, typical of a classical resistor. This is brought about by the growth in the meander resistance due to the increase in its temperature, which is caused by the heat released upon the electric current flow through the conductor according to Joule–Lenz's law. The coincidence of the forward and backward run of the curves in Fig. 3a is evidence of the reversibility of the process and the high rate of heating/cooling of the meander. No sluggishness of the heating at the chosen sweep rate of the supply voltage is observed.

With the use of the $I(U)$ dependence, we can calculate the resistance ($R = U/I$) of the heating element and the power consumed by it ($N = UI$) at various supply voltages. The resistance of the meander with a thickness of 300 nm increases regularly from 11.6 to 31.4 Ω in the course of the voltage sweep to 1.5 V (Fig. 3b). This takes place due to the increased scattering of the electrons on the phonons as a result of an increase in temperature.

Using the TCR value determined earlier by the experiment and starting from the measured $I(U)$ dependence, we can calculate the planar heating element temperature versus the supply voltage. For that, we should substitute the meander resistance obtained by Ohm's law ($U = IR$) into Eq. (1):

$$T = \frac{\left(\frac{U}{IR_R}\right) - 1}{\alpha} + T_R. \quad (3)$$

Here R_R is the heater's resistance at room temperature T_R. The dependence obtained using Eq. (3) is depicted in Fig. 3c. Note that in this way only the average meander temperature may be estimated under the assumption that the current-supplying leads and contact pads make a small contribution to the measured resistance.

The power consumed by the heater increases as the supply voltage is increased (Fig. 3d). However, its value, at typical supply voltages of about 1.2 V for 300-nm-thick platinum and 0.9 V for the 800-nm-thick series (which corresponds to a temperature of ~400°C), turns out to be lower than 52 and 90 mW, respectively. These values of the power consumption are appreciably lower than those for the existing bulk analogues (falling in the range from 100 to 300 mW for single heaters employed in sensors DTK-1 (Science and Technology Center of Measuring Gas Sensors, Russia), TGS6810 (Figaro Engineering Inc., Japan), NAP-100AC (Nemoto&Co Ltd., Japan), and 4p-50 CiTipel (City Technology Ltd., Great Britain).

Importantly the meander resistance rises linearly with temperature (Fig. 3e), which enables us to easily control the temperature of the active area of a planar heating element by measuring its hot resistance.

With an optical microscope in a darkened room, we can obtain visual corroboration of the heating of the active area of a planar heating element (Fig. 4). For the series of microheaters with a Pt thickness of 800 nm, the first signs of incandescence are seen at $U \approx 0.85$ V. As the voltage is increased, the incandescence intensity rises. The temperature corresponding to the red incandescence of the meander at 1.1 V is close to 480°C. Therefore, the voltammetry data and visual observations of the incandescence of the active area of the sensing element agree closely with each other.

Note that the subsequent increase in the supply voltage is inexpedient due to the large heat losses. In the steady-state mode, the heat fed due to electric heating is withdrawn by heat conduction, natural convection, and radiation. The convection heat transfer, according to Newton–Richman's law is proportional to the temperature difference at the interface between

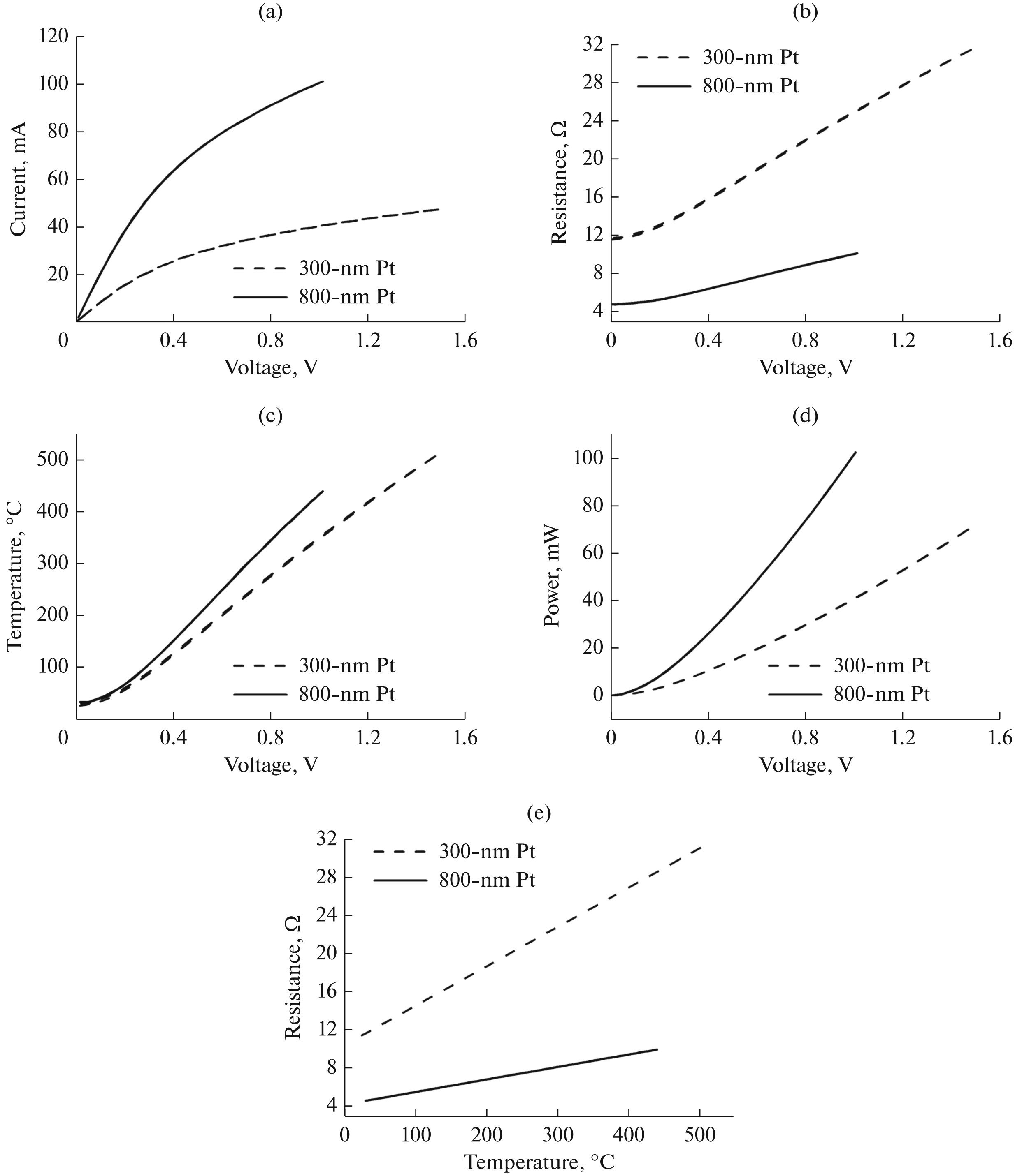

Fig. 3. Parameters of operation of planar heating element. (a) Current–voltage characteristic of elements with differing thicknesses and dependences of (b) resistance, (c) heater temperature, and (d) power consumption on supply voltage calculated from it. (e) Temperature dependence of resistance of meanders.

two media. When incandescence appears, the power withdrawn from the active area of a sensing element increases dramatically and becomes proportional to the temperature difference to the fourth power ($T_e^4 - T_a^4$), where T_e and T_a is the temperature of the sensing element and the ambient temperature, respectively [9].

Time Response of Planar Heating Element

To determine the time response of a planar heater of a thermocatalytic gas sensor, the samples were tested with the cyclic change of the operation mode (Fig. 5). With a stepwise increase in voltage (the interval from 2 to 3 s), the current rises sharply to 70 mA for

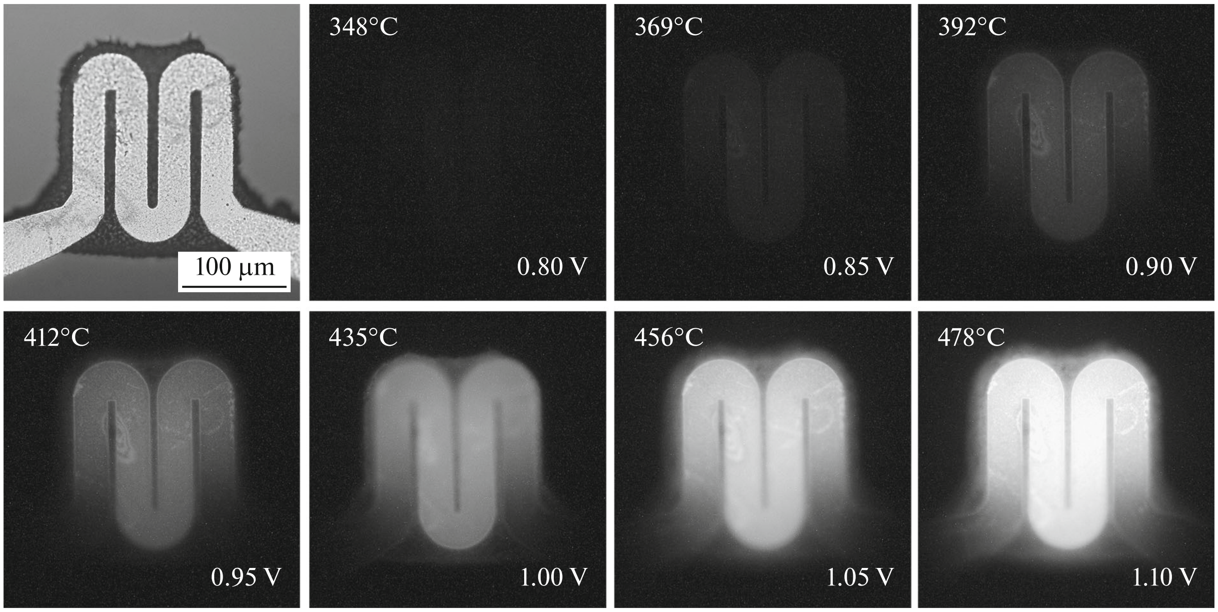

Fig. 4. Incandescence of planar heating element in dark at different supply voltages. Indicated temperature values are calculated using *I–V* characteristic and Eq. (3). In top left corner, photo of active area of sensing element in light is shown. Images are obtained by optical microscopy.

the 300-nm-thick heaters and 160 mA for the 800-nm-thick heaters. Further on, as the meander heats up (the resistance increases), the current diminishes and reaches a plateau corresponding to the steady-state operating mode. The reverse situation is observed when the supply voltage is decreased (the interval from 3 to 4 s): the current also does not stabilize immediately, due to the gradual cooling of the meander.

The quantitative characteristics of the time response, in particular, the time needed to achieve the steady-state mode, are shown in Table 1. The criterion for stationariness was the achievement of 90 to 99% of the heater's resistance in the steady-state mode. Note that the duration of the cooling process is much longer than the time required for heating.

The results indicate that a dynamic operating mode of the planar heating element is promising for use, which will make it possible in the future to decrease the power consumption of the average device by more than an order of magnitude.

Long-Term Stability of Operation

The stability of the resistance of a heating element, ensuring a constant temperature under a fixed supply voltage, is a compulsory requirement in the production of gas sensors. The long-term drift of the resistance of the heater was measured when it operated at a temperature of ~400°C. To minimize the influence of fluctuations in the ambient temperature, the microheaters were tested in a heating chamber at 40°C. The recorded $I(t)$ dependences under a constant voltage allow us to determine the drift of the resistance of the planar heating elements $\partial R/\partial t$. The experiments illustrate that, in the course of operation, there is a small increase in the resistance of the planar heater at a rate of 1.2% per month for elements with 300-nm-thick

Table 1. Time response parameters of planar heating elements with thicknesses of 300 and 800 nm on 30-μm-thick anodic alumina substrate. Ramp-up times (t_X) correspond to times needed for heater to reach resistance of *X*% of that in steady-state mode

Mode	Pt thickness, nm	Supply voltage, V	Power, mW	Ramp-up time, ms		
				t_{90}	t_{95}	t_{99}
Cooling from 400 to 100°C	300	0.33	6.9	107 ± 1	148 ± 1	248 ± 3
	800	0.28	15.2	87 ± 1	126 ± 1	236 ± 2
Heating from 100 to 400°C	300	1.23	51.1	43 ± 1	63 ± 1	121 ± 2
	800	0.92	88.5	34 ± 1	51 ± 1	108 ± 1

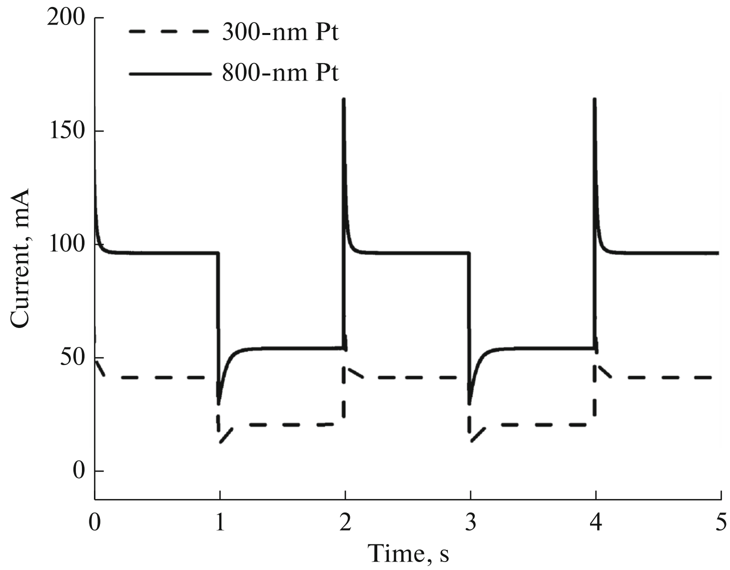

Fig. 5. Time response of planar heating elements with thicknesses of 300 (dashed curve) and 800 nm (solid curve). Tests were carried out in cyclic mode: operating temperature was ~400°C and standby temperature was ~100°C. Pulse duration was 1 s and rate of data acquisition was 1000 points per second.

platinum and an increase of less than 0.7% per month for meanders with 800-nm-thick platinum. Therefore, the obtained thin-film Pt microheaters on an anodic alumina substrate are fit for application for longer than 1 year.

Resistance to Impact Loads

The tests on resistance to the impact loads of the planar elements revealed their extraordinary stability. All the tested elements preserved their operating capacity after 5000 impacts. Note that for the pellistors that are currently widely used to detect inflammable gases in the atmosphere, the ability to operate after 1000 impacts is considered to be a very good characteristic. For the most part, bulk elements deteriorate significantly as soon as after 100 impacts.

Comparison with Analogues throughout the World

The creation of gas sensors based on planar sensing elements is extremely promising, and could result in a significant decrease in the cost of production and enhancement of their functional characteristics. As a result, in recent years, many companies specializing in the production of gas sensors have actively developed their planar elements. The main technical characteristics of the planar heating elements of different thicknesses reported here and their comparison with the existing analogues in the world are given in Table 2.

The CCS4_15 microheater, produced by CMOS Sensors, has comparable power and a higher rate of heating than those of the developed elements of 300-nm-thick platinum. This is achieved due to the small membrane thickness used by CMOS Sensors. At the same time, the microheaters obtained in this study are formed on a porous substrate with a thickness of 30 μm, which may serve as a support for a catalyst in the production of thermocatalytic gas sensors. In contrast, applying a catalyst support on a CCS4_15 microheater will significantly increase the mass of the active area and, therefore, increase the required power and weaken the response.

In the market, there are also gas sensors with a similar construction, a supply voltage of ~3 V and power consumption of 110 mW, with an operating temperature of 400 to 500°C [11]. We note that the alternative solutions for determining the concentration of volatile organic compounds [12] are operable at temperatures of ~300°C, which is insufficient for the thermocatalytic detection of inflammable gases (e.g., methane) and creation of semiconductor sensors.

CONCLUSIONS

In the study, a technique for the formation of planar heating elements of platinum on a porous anodic alumina substrate is proposed and successfully implemented. The resulting elements allow heating the active area to a temperature of 500°C and, at the same time, they have short response time and low power consumption. When operated in a steady-state mode at 400°C, the drift of the resistance of the platinum microheater with a thickness of 800 nm does not exceed 0.7% per month, which is sufficient for stable device operation for longer than 1 year.

Planar microheaters based on porous anodic alumina films, due to the uniform distribution of the mass

Table 2. Comparison of characteristics of planar heating elements of platinum with differing thicknesses reported here with current world analogues

	Present study		CMOS Sensors CCS4_15 [10]
	300-nm Pt	800-nm Pt	
Cold resistance, Ω	12.8 ± 0.9	4.26 ± 0.19	41 ± 3
Supply voltage at 400°C, V	1.23	0.92	~1.8
Temperature coefficient of resistance $\alpha \times 10^4$, 1/K	34.3 ± 0.7	27.8 ± 0.6	20.5
Power at 400°C, mW	~51	~88	~52
Maximum temperature, °C	~500	~500	~600
Heating duration t_{90}, ms	43 ± 1	34 ± 1	15 ± 5
Active area size, μm	150 × 150	150 × 150	200 × 200
Geometric dimensions, mm	3.5 × 4.5	3.5 × 4.5	0.9 × 1.0

over the product surface, exhibit extremely high stability to mechanical loads and can withstand more than 5000 impacts without breaking the electric circuit. This value is by an order of magnitude higher than the mechanical stability of pellistors. The resistance of planar elements to impact loads appreciably prolongs the service life of the sensing elements and reduces the costs of substituting them in portable gas alarms.

The reached characteristics of planar heating elements confirm the prospects of their application as a platform for semiconductor and thermocatalytic sensors of toxic and explosive gases. The synthetic approaches used in the work (anodic oxidation, photolithography, and magnetron sputtering) feature manufacturability and scaling simplicity, which ensures a low cost price and a high level of reproducibility of production.

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