A gas sensor based on zinc oxide obtained by magnetron spraying at direct current was investigated. There are methods of deposition of zinc oxide nanostructures such as thermal evaporation, chemical vapor deposition, organometallic chemical vapor deposition, magnetron sputtering, pulsed laser deposition, and hydrothermal process. The least investigated is magnetron sputtering. To obtain films, a vacuum unit VUP-5M with an original material-saving magnetron was used. Studies into the sensitivity and speed of the gas sensor based on ZnO with respect to the target gas – ethanol of different concentrations – were carried out. The resulting experimental dependences of the sensitivity of the gas sensor on the concentration of the target gas demonstrate that with increasing concentration of the target gas, the resistance decreased while the sensitivity of the sample increased. It was established that the change in the resistance of the test sample is proportional to the change in the concentration of the target gas. After the sensor surface becomes saturated with adsorbed molecules, the resistance no longer decreases, even if the gas concentration continues to increase. The reaction of the gas sensor to the target gas – ethanol – at concentrations above 150 ppm was almost absent. The time required to achieve the maximum response value should be lower at higher target gas concentrations. Sensitivity reaction repeatability studies were conducted to measure the resistance of a gas sensor based on ZnO in a target gas atmosphere with a concentration of 150 ppm. It was found that the gas sensor demonstrates excellent stability and consistent sensitivity reaction when re-exposed to the target gas – ethanol. It was established that the reaction time of a gas sensor based on ZnO to the target gas at each repeated exposure does not exceed 10 s. This repeatability index allows us to assert the stability of the ZnO-based gas sensor in an ethanol atmosphere under standard conditions.

***Keywords:** zinc oxide, gas sensor, magnetron sputtering, sensitivity reaction, target gas*

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IMPROVING THE MANUFACTURING TECHNOLOGY OF SENSING GAS SENSORS BASED ON ZINC OXIDE BY USING THE METHOD OF MAGNETRON SPUTTERING ON DIRECT CURRENT

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1. Introduction

In the context of the constant growth of threats to the population and the environment, especially as a result of hostilities, the problem of ensuring the stability of the functioning of various objects has become acute [1]. A special place is occupied by critical infrastructure objects [2, 3], which include almost all technical and environmental facilities [4, 5]. Destroyed oil depots, enterprises, factories, scorched forests, exploding ammunition, burning equipment, lubricants, diesel and gasoline pose a serious problem for the survival of living beings [6]. A separate category of hazards includes leakage of explosive and flammable gases [7]. Consequently, devices for monitoring the state of the gaseous environment in real time are extremely important for timely prevention of emergencies, protection of the public and the environment [8]. The popularity of gas analyzers is growing every year due to their various applications. Most often, gas analyzers are used to detect H_2 in fuel cells, CH_4 in mining, NO_2 in the automotive industry, hydrocarbons in oil refineries, NH_3 in the fertilizer industry [9, 10]. More and more branches of economic activity require the use of efficient, reliable, low-energy, highly sensing, and compact gas analyzers. To solve this urgent problem, gas analyzers based on metal oxides are promising due to their high sensitivity to many target gases in combination with simple manufacturing methods, their low cost, and compact size. Of particular interest are semiconductor oxides SnO_2, ZnO, In_2O_3, Ga_2O_3, TiO_2, WO_3, Fe_2O_3, CeO_2, and perovskites ($BaSnO_3$, $LaFeO_3$, $SrTiO_3$) with different morphologies [11]. The reactivity of the gas analyzer usually depends on the size of the nanostructures. The following methods are used to obtain different morphology of structures: chemical vapor deposition, radiofrequency spraying, hydrothermal, electrospinning, sol-gel method, template synthesis method, molecular beam epitaxy, and chemical vapor deposition. When using these methods, nanostructures such as thin films, nanoplates, nanospheres, nanowires, nanorods, nanotubes, nanoflowers, nanofibers, nanoneedles, and nanotapes are obtained [12]. Despite progress in the development of such gas sensors of various morphologies, some issues remain insufficiently studied. Among such issues, one should note the difficulties in constructing general mathematical models that would describe the operation of a gas sensor based on metal oxide. Models that have been developed are either very simplified or relate only to individual processes occurring during the operation of the gas sensor in the environment. Difficulties in constructing general mathematical models describing the operation of the sensor are due to a wide range of physical phenomena manifested in real conditions of the sensor. Experimental studies of sensors based on different materials and having different morphologies are not, as a rule, systemic. These circumstances introduce an element of uncertainty in the formation of directions for improving the efficiency of gas sensors based on metal oxides. To establish the dependences of sensor characteristics on the method of obtaining and, accordingly, the morphology of the structure, further research is needed.

2. Literature review and problem statement

Zinc oxide is a direct-gap semiconductor with a large band gap (3.37 eV) and a large exciton binding energy (60 meV) [8], which is a significant advantage among other metal oxides. The simple and inexpensive synthesis of ZnO, together with the response value of the pure material, compared to the performance of commercially available materials, make it one of the most researched materials for use as a gas sensor [8]. Since ZnO is a chemoresistive sensor, the change in its resistance depends on the presence of chemisorbed oxygen ions. Oxygen molecules are adsorbed on the surface of ZnO in the presence of atmospheric air. Thus, their formation occurs due to the extraction of electrons from the ZnO conduction band, which increases the resistance to ZnO. If reducing gases interact with chemisorbed oxygen ions on the ZnO surface, there is a decrease in resistance since oxygen ions donate free electrons to the ZnO conduction band.

In particular, the higher the effective surface area, the higher the adsorption of the contamination molecule [13]. However, the question of optimal structure morphology for stable sensor operation requires further research. Among other types of morphologies, ZnO nanorods are of particular interest due to their high surface-to-volume ratio [14], in addition, such structures are easily synthesized. In [15] it is shown that the reaction of a gas sensor based on ZnO is significantly influenced by operating temperature and gas concentration. The authors obtained the dependence of the reaction of the sesor on temperature but the issue of operation of the gas sensor under standard conditions was not solved. In [16], it is described that the reaction of the sensor is reduced due to a decrease in chemisorbed oxygen ions on the surface above the optimum operating temperature. Therefore, the operating temperature of the gas detector is kept as low as possible but when the operating temperature drops to standard, the efficiency of the gas sensor decreases. In [17, 18], ZnO nanorods were chemically manufactured on aluminum foil at low temperature. The chosen synthesis method is inexpensive and simple for the manufacture of various nanostructures. The resulting gas sensor demonstrated a stable reaction to liquefied gas. When studying the sensitivity of a sensor based on zinc oxide, it was found that such a sensor is effectively used for quantitative measurements of liquefied gas, for example, reducing gases. In [19], ZnO nanowires and ZnO nanowires with Pd-coating, which were obtained by hydrothermal method, were investigated. Both types of nanowires showed a high value of selectivity to ethanol, but the sensitivity of gas sensors was recorded only at temperatures above 325 °C. In [20], nanorods based on ZnO with Pd-coating for ethanol measurement were investigated. The research results showed that the response and recovery time is 14 s and 70 s, respectively, under the influence of ethanol with a concentration of 1530 ppm at a temperature of 200 °C. Despite significant improvements in sensitivity, the recovery time of the investigated sensors remains long. The use of Pd coatings has improved the sensor performance based on ZnO, namely its sensitivity and response. However, the developed sensors do not have long-term stability and demonstrate a long recovery time. In [21], ZnO-based sensors with sensitivity and selective response to 1.2 ppm of ethane and ethylene were obtained. Such results were obtained by irradiating samples with gamma radiation. It was found that porosity, crystallite size, and point defects (mainly oxygen and zinc vacancies) of the studied ZnO thin films increase with increasing gamma radiation. Accordingly, there was an increase in sensitivity with an increase in the radiation dose. However, this approach to the creation of gas sensors complicates the process of large-scale production. Among the various methods of ZnO nanostructure deposition, the magnetron sputtering method is the least studied.

At the same time, magnetron sputtering is most widely used for uniform deposition of thin films. Depending on the growing conditions, the upper surface of ZnO nanostructures ends up with zinc or oxygen ions. This predetermines potential applications at room temperature. In addition, it has recently been reported that defects in nanograined ZnO significantly alter its physical and electronic properties.

Thus, the need to improve sensitivity response, rapid response, long-term stability, selectivity, and reproducibility at low operating temperatures leads to the search for structural and technological solutions for ZnO-based gas sensors.

3. The aim and objectives of the study

The aim of this work is to improve the technology of creating sensing gas sensors based on zinc oxide. This will make it possible to design semiconductor gas sensors using simple methods suitable for large-scale production.

To accomplish the aim, the following tasks have been set:

– to study the sensitivity of a gas sensor based on ZnO, obtained by magnetron sputtering on direct current;

– to study the stability of the sensitivity reaction of a gas sensor based on ZnO, obtained by magnetron sputtering on direct current.

4. The study materials and methods

4. 1. Preparation of nanostructured zinc oxide by magnetron sputtering on direct current

We obtained the studied samples of a gas sensor based on ZnO by magnetron sputteringing on direct current. To obtain films, a vacuum unit VUP-5M (Fig. 1) with an original material-saving magnetron was used (Fig. 2).

Fig. 1. Vacuum unit VUP-5M

Fig. 2. Material-saving magnetron for vacuum unit VUP-5M

To form a zinc target (99.99 % purity), a special press was used (Fig. 3). To determine the thickness of the films, a helium-neon laser with a wavelength of 633 nm was used.

Fig. 3. Zinc target molding press

The length of the discharge gap, which is the distance between the magnetron and substrate, was 70 mm. The magnetron power was 0.2 W/cm^2. High purity argon was used as an inert gas, oxygen as an active gas. The output pressure in the vacuum chamber was 3×10^{-5} mmHg, the working pressure of the argon-air mixture during spraying was $(2.1–2.6)\times10^{-2}$ mmHg. The substrate temperature was 300 °C. The deposition rate was 12 Å/s. The resulting films had a thickness of 2 microns.

4. 2. Investigation of sensitivity and performance of the gas sensor based on ZnO

The sensitivity study of the gas sensor based on ZnO was carried out using an installation, the block diagram of which is shown in Fig. 4.

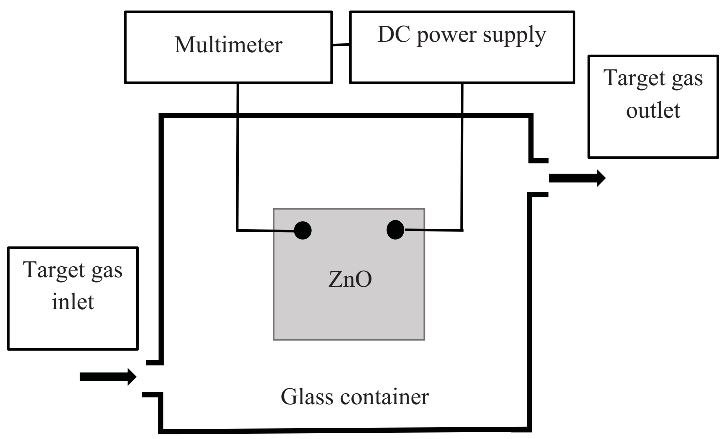


Fig. 4. Block diagram of the installation for studying the sensitivity of the gas sensor

The measuring unit consisted of a constant voltage source, a digital multimeter, and a controller. The test sample of a gas sensor based on ZnO was placed in a glass box with a volume of 3.6×10^{-2} m^3. A constant voltage source provided a voltage between the two electrodes of 5 V. The current flowing through the gas sensor was measured using a multimeter. Ethanol (C_2H_6O) was supplied as the target gas. The glass box was filled with the test gas by an air pump. The concentration of the target gas was regulated using speed flow and feed time at ambient temperature. The relative humidity was maintained at 50 %. The concentration of the target gas varied from 50 ppm to 150 ppm. To obtain experimental values, 12 parallel experiments were conducted.

To determine the sensitivity of the gas sensor, an air-water mixture is supplied to the measuring unit chamber until a constant value of the sensor signal measured using a multimeter is established. After that, the target gas is supplied to the chamber until a certain concentration is established. After setting a constant signal value, the value of the multimeter is recorded. Measurements are repeated for each of the selected concentrations. The sensitivity value (S) was calculated as:

$$S=\frac{R_g-R_0}{R_0}, \qquad (1)$$

where R_0 is the resistance of the gas sensor in the air, R_g – resistance of the gas sensor in the atmosphere of the target gas.

To establish the speed of the gas sensor, an air-water mixture is supplied to the measuring unit chamber until a constant value of the sensor signal measured using a multimeter is established. After that, the target gas is supplied to the chamber every 10 seconds. The reading of the multimeter is recorded before the establishment of a constant signal scale.

5. Results of the study of nanostructured ZnO intended for use as a gas sensor

5. 1. Results of investigating the sensitivity of the gas sensor based on ZnO obtained by magnetron sputtering

According to the results of the study of changes in the resistance of the gas sensor, we plotted a sensitivity dependence of the test sample on target gas concentration. Fig. 5, *a–c* demonstrates sensitivity dependences at a concentration of the target gas of 50 ppm, 100 ppm, 150 ppm.

Given that oxygen from atmospheric air is adsorbed on the surface of ZnO and pushes electrons out of the conduction band to form O_2 on the surface [22]. After the gas sensor enters the atmosphere of the target gas, the target gas (TG) reacts with oxygen adsorbed on the surface of ZnO (O_{ads}). This leads to the formation of a compound (TGO) and the release of electrons from oxygen ions into the conductive zone of the gas sensor, which leads to a decrease in the resistance of the test sample:

$$TG+O^{2-}\rightarrow TGO+2e^-. \qquad (2)$$

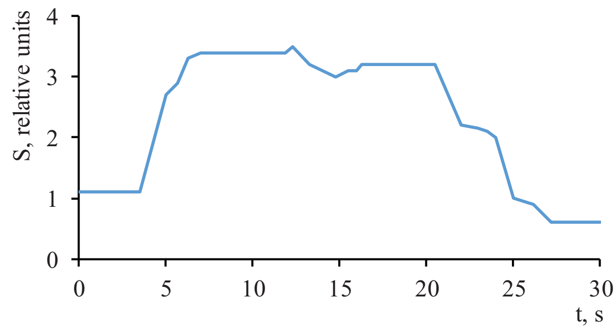
In the case of formation on the surface of O^{2-}, the concentration of charge carriers (N) on the surface will be less than with a single ionosorption of the gas sensor. At lower concentrations of charge carriers, the sensitivity of the sensor increases. Once the expression for determining the sensitivity of gas is written through conductivity (p), then we obtain the following:

$$p=Ne\mu, \qquad (3)$$

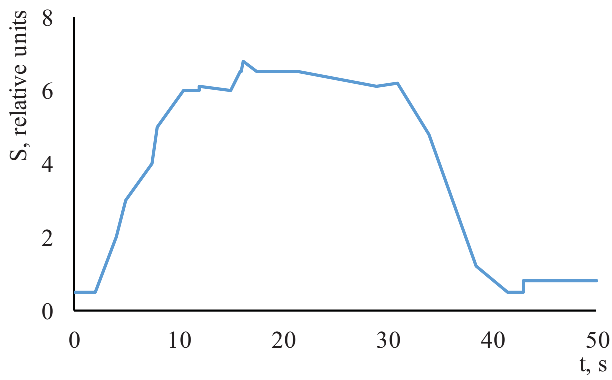
where N is the concentration of charge carriers, e – electric charge, μ – molar mass:

$$S=\frac{p_0-p_g}{p_g}=\frac{\Delta p}{p_g}, \qquad (4)$$

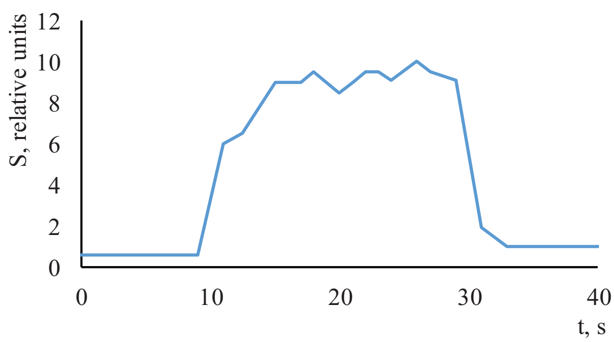
where p_0 is the conductivity of the gas sensor in the air, p_g – conductivity of the gas sensor in the atmosphere of the target gas.



a



b



c

Fig. 5. Sensitivity of ZnO-based gas sensor to target gas at the following concentration: *a* – 50 ppm; *b* – 100 ppm; *c* – 150 ppm

In this case, the sensitivity of the gas sensor can be represented by the ratio:

$$S=\frac{\Delta p}{p_g}=\frac{\Delta N}{N}. \qquad (5)$$

Thus, we can conclude that the sensitivity of the gas sensor is proportional to the change in the concentration of charge carriers.

According to the results of the study into the influence of the concentrations of target gas on the resistance of the investigated gas sensor based on ZnO over time, we built the dependences shown in Fig. 6.

The results of the study of reaction time and recovery of the gas sensor based on ZnO are shown in Fig. 7.

As can be seen from Fig. 7, the response time of the ZnO-based gas sensor decreases with increasing concentration of the target gas while the recovery time increases.

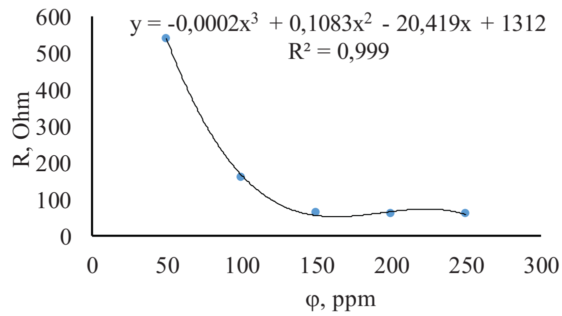


Fig. 6. Resistance of gas sensor of ZnO-based gas sensor to target gas

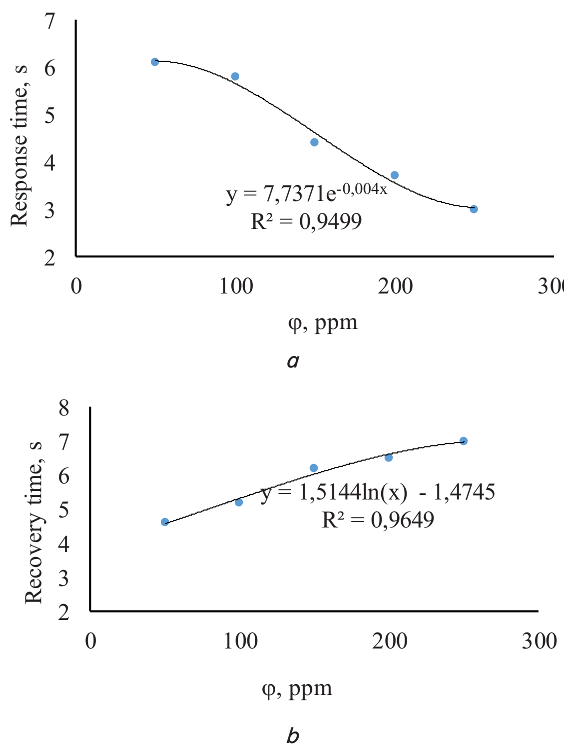


Fig. 7. The reaction of a gas sensor based on ZnO to the target gas of different concentrations: *a* — response time; *b* — recovery time

5. 2. Results of investigating the reaction stability of a ZnO-based sensor obtained by magnetron sputtering

To analyze the stability of the reaction of the sensitivity of the gas sensor based on ZnO to the target gas, we built the dependences shown in Fig. 8.

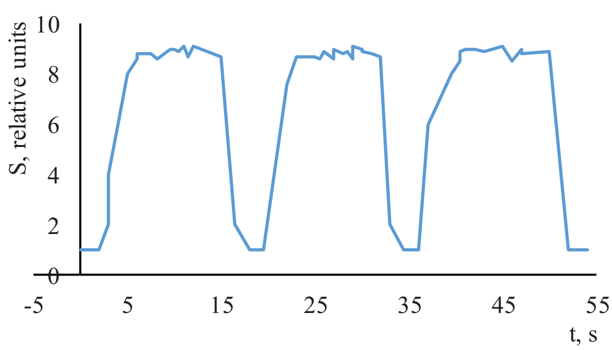


Fig. 8. Reaction of ZnO-based gas sensor sensitivity to target gas under repeated exposure (gas concentration, 150 ppm)

Sensitivity reaction repeatability studies were conducted to measure the resistance of a gas sensor based on ZnO in a target gas atmosphere with a concentration of 150 ppm.

6. Discussion of results of investigating the nanostructured ZnO obtained by magnetron sputtering on direct current

The analysis of dependences (Fig. 5, *a–c*) demonstrates that the change in the resistance of the test sample is proportional to the change in the concentration of the target gas. Thus, we can conclude that the sensitivity of the gas sensor is proportional to the change in the concentration of charge carriers. This behavior was observed when the ZnO-based gas sensor was in the atmosphere of the target gas, as shown in Fig. 6. Higher gas concentrations increase the absorption of gas molecules on the surface of the gas sensor, which leads to lower resistance. After the sensor surface becomes saturated with adsorbed molecules, the resistance no longer decreases, even if the gas concentration continues to increase. The resistance of the ZnO-based gas sensor was stable when the gas concentration was above 150 ppm.

The concentration of the target gas affects the number of molecules adsorbed on the surface of the gas sensor, which determines the response and recovery time of the sensor. Higher gas concentrations contribute to the absorption of the target gas, which increases the sensor response rate. Therefore, the time required to achieve the maximum response value should be lower at higher target gas concentrations. This behavior of the gas sensor was observed, as shown in Fig. 7, *a*. However, the more molecules are adsorbed on the surface, the longer it takes to release these molecules. Therefore, the recovery time increases at higher concentrations of the target gas, as shown in Fig. 7, *b*.

Our results from the ZnO-based gas sensor study on repeatability and stability of operation demonstrated that the reaction of the sensitivity of the ZnO-based gas sensor to the target gas is repeated. That is, a gas sensor based on ZnO demonstrates excellent stability and consistent sensitivity reaction with repeated exposure to the target gas (Fig. 8).

Studies of the gas sensor were carried out under standard conditions, changing the sensitivity of the gas sensor to the target gas under conditions of low autosphere air temeratures requires further research. The sensitivity of the gas sensor to ethanol in the gas mixture has not been determined.

Our results of the ZnO-based gas sensor study on repeatability and stability of operation demonstrated that the reaction time of the gas sensor to the target gas of fixed concentration almost does not increase with repeated exposures. The reaction time of the ZnO-based gas sensor to the target gas at each repeated exposure does not exceed 10 s. This repeatability index allows us to assert the stability of the ZnO-based gas sensor in an ethanol atmosphere under standard conditions.

The study of gas sensors based on ZnO, obtained by magnetron spraying on direct current, showed that the obtained instrument structures can be used at a target gas concentration of more than 50 ppm. At lower concentrations of ethanol, a change in the resistance of the semiconductor layer is not observed. Thus, limitations on the operation of gas sensors based on ZnO, obtained by magnetron spraying on direct current, include the threshold of sensitivity to ethanol at 50 ppm. Studies of the gas sensor were carried out under standard conditions, changing the sensitivity of the gas sensor to the target gas at low ambient temperatures requires further research. The shortcomings of our studies include the lack of results on the selectivity of the obtained gas sensors. It is the selectivity that determines the consumer qualities of the sensor. Further studies of gas sensors based on ZnO, obtained by magnetron spraying on direct current, will focus on determining the selectivity of instrument structures.

7. Conclusions

1. We have studied the sensitivity of the gas sensor based on ZnO, obtained by magnetron spraying on direct current. It was found that the change in the resistance of the test sample is proportional to the change in the concentration of the target gas. After the sensor surface becomes saturated with adsorbed molecules, the resistance no longer decreases, even if the gas concentration continues to increase. The reaction of the gas sensor to the target gas, ethanol, at concentrations above 150 ppm was almost absent. The time required to achieve the maximum response value should be lower at higher target gas concentrations.

2. The studies of stability of sensitivity reaction of gas sensor based on ZnO obtained by magnetron spraying on direct current have been carried out. It was found that the gas sensor demonstrates excellent stability and consistent sensitivity reaction when re-exposed to the target gas – ethanol. It was established that the reaction time of a gas sensor based on ZnO to the target gas at each repeated exposure does not exceed 10 s. This repeatability index allows us to assert the stability of the ZnO-based gas sensor in an ethanol atmosphere under standard conditions.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Data availability

The data will be provided upon reasonable request.

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