



## Section 3. Chemistry

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### SELECTION OF HIGHLY ACTIVE MATERIAL FOR SEMICONDUCTOR SENSORS OF CARBON MONOXIDE, HYDROGEN AND HYDROCARBONS BASED ON INDIVIDUAL METAL OXIDES

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#### Abstract

In this work, we investigated the catalytic activity of individual metal oxides (CuO, MnO<sub>2</sub>, SnO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>, Co<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, TiO<sub>2</sub>, etc.) in the oxidation of carbon monoxide (CO), hydrogen (H<sub>2</sub>), and hydrocarbons (methane, n-octane) in order to select the optimal material for semiconductor gas sensors. Experiments were conducted using gas-air mixtures at a controlled temperature of 350 °C and a fixed flow rate. The activity and selectivity of the oxides, as well as their dependence on physical properties, such as the metal–oxygen bond length and band gap, were determined. The results show that CuO, MnO<sub>2</sub>, and SnO<sub>2</sub> are the most active oxides for CO oxidation, and ZrO<sub>2</sub> is the most active for the selective detection of H<sub>2</sub>. These findings enable the creation of semiconductor sensors with improved operational and metrological characteristics.

**Keywords:** semiconductor sensor, carbon monoxide, hydrogen, hydrocarbons, metal oxides, catalytic activity, selectivity

#### Introduction

Semiconductor sensors are widely used to monitor toxic and flammable gases, such as CO, H<sub>2</sub>, and hydrocarbons, in industry

and households. The main challenge in developing such sensors is selecting an active material with high catalytic activity and selectivity for the target gases. Metal oxides are

promising candidates due to their high stability, catalytic activity, and the ability to fine-tune their properties.

The aim of this work is to identify the most active and selective individual metal oxides for the development of semiconductor sensors capable of reliably detecting CO, H<sub>2</sub> and hydrocarbons in gas mixtures with different component concentrations.

### Methodology

**Selection of material:** metal oxides CuO, MnO<sub>2</sub>, SnO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>, Co<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, TiO<sub>2</sub>, WO<sub>3</sub>, NiO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> were selected as the objects of study.

**Catalyst preparation:** The oxides were deposited on an inert support (Al<sub>2</sub>O<sub>3</sub> or SiO<sub>2</sub>) using the sol-gel method to form an active catalytic layer ~1 mm thick. For some catalysts, Pt or Pd were added to improve activity.

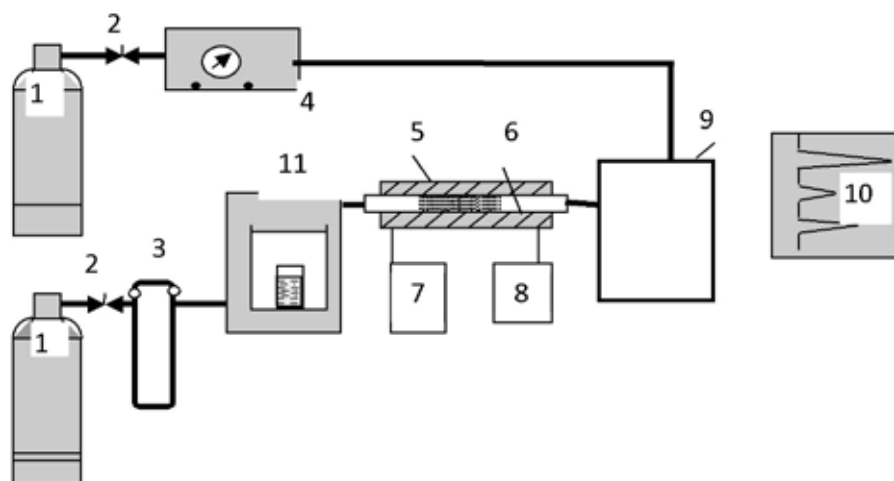
**Performance control:** The physical properties of the materials – metal–oxygen bond length, band gap, and particle size – were taken into account when analyzing the results.

**Activity determination:** oxidation of CO, H<sub>2</sub> and hydrocarbons was carried out at 350 °C with a fixed flow of gas-air mixture (10 ml/min) and known partial pressure of the components.

In order to develop sensors that monitor carbon monoxide, methane and hydrocarbons (gasoline vapors, natural gas and others) in gas and gas-vapor mixtures, the oxidation processes of these substances in the presence of various metal oxides were studied.

The experiments were carried out in a reactor with a constant catalyst bed, the diagram of which is shown in Figure 4.1.

**Figure 1.** Scheme of the installation for catalytic oxidation of combustible substances



- 1 – gas cylinders;
- 2 – fine gas regulation valve;
- 3 – rheometer;
- 4 – gas preparation device;
- 5 – electric furnace;
- 6 – reactor with catalyst;

- 7 – current source;
- 8 – millivoltmeter;
- 9 – chromatograph;
- 10 – recording device KSP-4;
- 11 – source of the mixture being studied (gas or steam)

The main elements of the installation include: a reactor with a catalyst, a device for preparing and feeding gases, a chromatographic dispenser and a gas chromatograph.

### Experimental part

The reactor is a heat-resistant glass tube equipped with an electric furnace. The required temperature in the reactor is main-

tained with an accuracy of  $\pm 1.0$  °C using a thermostat. Temperature changes are monitored by a thermocouple connected to a millivoltmeter.

During the experiment, the gas-air mixture from the cylinder is fed through the rheometer into the reactor.

After the reaction mixture passes through the catalyst bed in the reactor, a certain

portion of this mixture is periodically directed into the chromatographic column using a metering valve.

The carrier gas from the cylinder passes through a fine-tuning regulator, a gas preparation unit (GPU), and a metering valve before entering the chromatography column. When a sample is injected, the carrier gas flow passes through the metering valve ring and captures a certain portion of the gas-air mixture.

The degree of oxidation of the combustible component is monitored by recording chromatograms of the mixture before and after it passes through the catalyst layer.

Chromatographic analysis of a gas mixture (carbon monoxide, hydrogen, methane mixed with air) is carried out under the following conditions:

- detector – katharometer;
- detector and column temperature – room temperature;
- carrier gas flow rate – 1.5–3.5 l/h (depending on the composition of the gas-air mixture);
- column length – 1.5–3 m, internal diameter – 3 mm, filling – activated carbon.

The quantity of components is determined using a pre-constructed calibration curve.

An LHM-8MD chromatograph with a flame ionization detector was used to monitor hydrocarbon (gasoline vapor) oxidation processes. The following conditions were used for chromatographic monitoring of gasoline and diesel fuel oxidation products:

- a chromatographic column filled with 5% apieson-L on Chromaton, 1 m long and 3 mm internal diameter;
- column temperature – 150–200 °C; the speed of transporting argon is 50 ml/min.

Additional control of the hydrocarbon oxidation process was carried out by determining carbon dioxide in the reaction products using potentiometric titration.

The completeness of oxidation of the component being determined was used as a criterion for the suitability of the selected catalyst for creating the sensing element of a semiconductor sensor. Considering that the completeness of oxidation of combustible substances depends on their composition, process temperature, the concentration

of reactants, and the ratio of components in the gas mixture passing through the reactor, we examined the role of each of these factors during the study.

Typically, catalysts used for the oxidation of hydrogen, carbon monoxide, methane and hydrocarbon vapors are noble metals, alloys, metal oxides and their mixtures, as well as systems based on platinum group metals.

As follows from the literature, oxidation reactions of combustible gases on Pt- and Pd-based catalysts have been studied in considerable detail. Such studies are considerably less widespread in the field of semiconductor materials. Among such compounds (oxides, sulfides, chlorides, etc.), rare metal oxides exhibit the highest activity, since, according to coordination theory, the presence of free coordination sites in the cationic sphere is necessary for the catalytic reaction to occur. Chlorides and sulfides ( $\text{Cl}^-$  and  $\text{S}^{2-}$ ) have a radius larger than oxygen and completely shield the cation, resulting in a significant reduction in catalytic activity.

Metal oxides exhibit significantly higher catalytic activity in the oxidation of CO,  $\text{H}_2$ ,  $\text{CH}_4$ , and other gases, which is why they are widely used in gas masks and in industrial and automotive exhaust gas purification systems. In combustible gas oxidation reactions, oxides are significantly more stable than metals, as they are stable in the presence of oxygen.

Along with activity and stability, one of the most important characteristics of a catalyst is its selectivity – the ability to accelerate only one reaction out of several occurring simultaneously. Therefore, the problem of selectivity in catalytic processes is of fundamental importance. It should be noted that when studying the oxidation of combustible substances, researchers primarily focus on the selection of active catalysts. Issues of catalyst selectivity have been much less studied. Moreover, due to the incomparability of data obtained for different catalysts under different conditions, it is impossible to draw even tentative conclusions about the selectivity of the oxidation of individual substances in the presence of other combustible gases and vapors. Developing a scientific basis for catalyst selection is one of the key tasks of catalysis. However, to date, a satisfactory theory of selectivity has not been developed.

Existing theories of catalysis – electron, multiplet, intermediate compound theory, and acid-base catalysis – define a number of properties of a solid that suggest its selective action in a given reaction, but none of these properties can be considered an exact criterion for catalytic selectivity. Therefore, the selection of active and selective catalysts is currently primarily accomplished experimentally. However, advances in the study of chemisorption and the mechanism of catalysis make it possible to significantly reduce the time required to select a catalyst for a specific reaction and identify a number of patterns.

Based on the above, the main objective of the research devoted to the development of a semiconductor gas sensor is the creation of selective catalytic systems with improved performance properties and metrological characteristics.

The catalyst for a sensor designed to detect natural gas and hydrocarbon vapors in industrial and automotive emissions was selected in the presence of hydrogen, carbon monoxide, methane, and other gases that occur together with hydrocarbons in various sources.

Aluminum and silicon oxides exhibit low activity in oxidation-reduction reactions. Therefore, we used aluminum oxide and silicon oxide as an inert substrate and as a carrier for the immobilization of active metal oxides. Formation of a catalytically active layer on the surface of the inert carrier was achieved using sol-gel technology.

To test the catalytic properties, the metal oxide was applied to an inert carrier by treatment with solutions of the corresponding metal salts (nitrates, carbonates or oxalates), followed by drying at 100–130 °C for 2.5–3 hours and calcination at the salt decomposition temperature for 3 hours.

Catalysts containing platinum and palladium were prepared by impregnating the carrier with aqueous solutions of chloroplatinic and chloropalladic acids, then drying at 130 °C and calcining at 500–700 °C.

Experiments to select optimal conditions for the oxidation of CO, H<sub>2</sub> and hydrocarbons in the presence of metal oxides were carried out at a gas-air mixture flow rate of 5 l/h and in a wide range of concentrations of combustible components. In semiconductor sensors, the active components of the gas-sensitive

film ensure the oxidation of combustible gases predominantly in a thermodynamically favorable direction with the formation of carbon dioxide and water vapor.

Experiments on the selection of active and selective metal oxides for gas-sensitive films of a semiconductor sensor designed to detect hydrogen, methane, carbon monoxide and hydrocarbon vapors were carried out using the following oxides: Fe<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub>, TiO<sub>2</sub>, ZnO, SnO<sub>2</sub>, WO<sub>3</sub>, ZrO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>, CuO.

Reaction conditions: catalyst layer in the reactor was 5.0 mm, the rate of gas-air mixture passage through the catalyst layer was 10 ml/min, samples were taken every 10 minutes for reaction mixture analysis, the volume of each sample was 0.1 ml. Catalysts were prepared on the basis of metal oxide powders, particle size ~1 mm. The partial pressure of the reducing component (CO, CH<sub>4</sub>, n-octane and H<sub>2</sub>) in the gas-air mixture for all components was: P<sub>com.</sub> = 0.02 atm., PO<sub>2</sub> = 0.2 atm., P<sub>atm.</sub> = 1.0 atm.

The composition of the reaction mixture before and after the catalyst layer was monitored using gas chromatography. The quantities of individual components during analysis were determined using pre-constructed calibration curves for each component.

The table shows that the most easily oxidized compound in the carbon(II) oxide-methane-n-octane-hydrogen system is hydrogen. For most of the metal oxides studied, the oxidation state of hydrogen is 50–100%.

When determining hydrogen, the highest selectivity is observed for zirconium oxide: in its presence, the oxidation of hydrogen reaches 100%, while the oxidation of carbon(II) oxide, methane and n-octane is, respectively: CO – 6%, CH<sub>4</sub> – 2.1%, n-octane – 0.9%.

These table results confirm the feasibility of using ZrO<sub>2</sub> as the main component of a gas-sensitive film in a semiconductor sensor that selectively detects hydrogen in the presence of carbon monoxide and hydrocarbons. Moreover, with this gas-sensitive material, carbon monoxide and hydrocarbons are virtually not oxidized under identical conditions.

According to the data in Table 3.1, at a temperature of 350 °C, all of the catalysts studied exhibited carbon monoxide oxidation to varying degrees. The most active metal oxides for this process include CuO, MnO<sub>2</sub>,

SnO<sub>2</sub>, and Cr<sub>2</sub>O<sub>3</sub>. In the presence of these oxides, CO oxidation under the experimental conditions ranged from 70 to 91%.

Oxides exhibiting moderate activity for the carbon monoxide oxidation process include Fe<sub>2</sub>O<sub>3</sub>, CoO, Co<sub>3</sub>O<sub>4</sub>, V<sub>2</sub>O<sub>5</sub>, ZnO, and TiO<sub>2</sub>. These oxides provided CO oxidation in the range of 40–68%.

The oxides with low activity and selectivity among the studied compounds were WO<sub>3</sub>, NiO, ZrO<sub>2</sub>, SiO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub>. At a temperature of 350 °C, carbon(II) oxide was practically not oxidized in the presence of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>; its oxidation by atmospheric oxygen amounted to only 2–3%.

Therefore, in subsequent experiments, these oxides were used as an inert carrier in the development of gas-sensitive elements of

semiconductor sensors for carbon monoxide monitoring.

### Conclusion

Individual metal oxides exhibit different catalytic activities and selectivities in the oxidation of CO, H<sub>2</sub> and hydrocarbons. For semiconductor CO sensors, CuO, MnO<sub>2</sub> and SnO<sub>2</sub> have the best activity. For selective detection of H<sub>2</sub>, ZrO<sub>2</sub> is optimal, as it ensures complete oxidation of hydrogen with minimal impact on other components. The highest catalytic activity in the oxidation of hydrocarbons (CH<sub>4</sub> and n-octane) is observed on Co<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, CuO and MnO<sub>2</sub>. The obtained data enable the development of highly efficient and selective semiconductor sensors for monitoring toxic and flammable gases in industrial and environmental systems.

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