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From Titanium Foil to Gas Sensor: Nanoporous Anodic Titania as a Functional Material

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Abstract We present a gas sensor using nanoporous titanium dioxide which is fabricated by anodic oxidation of a titanium foil. The process is easy to handle, can be carried out at low cost and the sensor response to reducing gases can be measured using a cheap digital multimeter. In contrast to the most gas sensors, no commercially available sensor substrate is required. The band model for semiconductors and a model of ionosorption provides an explanation for the working principle of the gas sensor on particle level. Several experimental approaches for upper-secondary chemistry class are presented.

Keywords: *gas sensor, titanium dioxide, band model*

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1. Introduction

Semiconducting metal oxides for gas sensing have already been widely investigated. Back in 1962, Seiyama et al. discovered that a film consisting of zinc oxide as an *n*-type semiconductor changes its electrical surface resistance dependent on the presence of oxidizing and reducing gases [1]. Afterwards, many more metal oxides were used and investigated for gas sensing applications, e.g. tin dioxide (SnO_2) and titanium dioxide (TiO_2) [2,3,4]. As an improvement of existing gas sensors, heterostructures made from TiO_2/V_2O_5 [5] and $TiO_2/Ag_{0.35}V_2O_5$ [6], Ce-doped SnO_2 [7] and sensors using $LaFeO_3$ [8] were developed. Especially titanium dioxide as a nanostructure has come into interest of researchers for gas sensing purposes. As the reactions on a gas sensor usually take place on the metal oxide surface, it is advantageous to enlarge the reactive surface area, e.g. by fabricating a nanoporous structure. Such nanostructures can be synthesized electrochemically by anodic oxidation [9].

For educational purposes, gas sensors based on several metal oxides have rarely been investigated. In the late 1980s, Bretschneider et al. detected emissions of combustion engines with a gas sensor [10]. By using an electronic gas sensor, Zeiter detected oxygen produced by photosynthesis [11]. In 2009, we used sensor substrates which are commercially available and developed several experiments for high school. A band model for semiconductors combined with a model of ionosorption provides an explanation for the sensor resistance changing in presence of oxidizing and reducing gases [12].

In this work, we present several gas sensing experiments for upper-secondary chemistry class using nanoporous titanium dioxide which is fabricated by anodic oxidation. Due to the direct contact with conductive titanium metal, no additional and cost intense sensor substrate is required. Furthermore, the anodic oxidation process ensures a nanoporous structure without the need to handle potentially hazardous nanopowders. In addition to the presented experiments, we show an outlook on the potential of gas sensing for chemistry class.

2. Fundamentals

In this section, the anodic formation of titanium dioxide layers in general as well as the introduction of porosity are presented. Furthermore, the working principle of semiconductor gas sensors is summarized.

2.1. Fabrication of Nanoporous Titanium Dioxide Layers by Anodic Oxidation

Anodic oxidation is a method to easily fabricate metal oxide layers and is widely used for common materials such as aluminum and titanium which will be used here [13]. At the cathode, gaseous hydrogen is released by the reduction of protons (equation 1) [13][14].

$2\ H^+ + 2\ e^- \rightarrow H_2 \uparrow$ (1)

At the anode, according to equation 2, titanium metal is oxidized and reacts with water to titanium dioxide [15].

$Ti + 2\ H_2O \rightarrow TiO_2 + 4\ H^+ + 4\ e^-$ (2)

Formally, this reaction can be described as a combined process: on the one hand, titanium(IV)-ions are formed (equation 3); on the other hand, titanium(IV)-ions react with water on the electrode surface to titanium dioxide which forms dense compact layers on the surface of the electrode (equation 4) [15].

$$Ti \rightarrow Ti^{4+} + 4\ e^{-} \qquad (3)$$

$$Ti^{4+} + 2\ H_2O \rightarrow TiO_2 + 4\ H^{+} \qquad (4)$$

To provide nanoporosity, an electrolyte containing fluoride ions must be used. In this case, during the anodic oxidation of titanium dioxide, a hexafluoridotitanate(IV)-complex is formed by the partial dissolution of titanium dioxide (equation 5) [16][17].

$$TiO_2 + 6\ F^{-} + 4H^{+} \rightarrow [TiF_6]^{2-} + 2\ H_2O \quad (5)$$

Due to the continuous formation and dissolution of titanium dioxide and a lower pH value at the bottom of the tubes leading to a higher dissolution rate of titanium dioxide, nanotubes are formed [17]. The process leading to the formation of nanotubes is described in detail in [18].

2.2. Working Principle of a Metal Oxide Gas Sensor

Titanium dioxide, like some other metal oxides, is a semiconducting material. Its conduction properties can therefore be explained using the band model. Additionally, the operation of a gas sensor is based on the change of electrical resistance due to ionosorption on the surface. Hence, the band model and the model of ionosorption need to be combined [12].

In the first step, atmospheric oxygen is adsorbed on the titanium dioxide surface. By accepting electrons from the conduction band, reactive oxygen species (e.g., O^{2-}, O^{-}, O_2^{2-}) are formed (ionosorption). Figure 1 shows the process of adsorption on the metal oxide surface and the formation of different oxygen species.

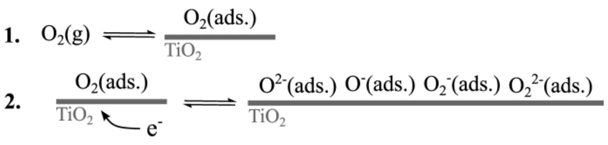

Figure 1. Adsorption of oxygen on the metal oxide surface (1) and formation of different oxygen species (2) [12].

In a next step, reducing gases can react with the adsorbed oxygen species at elevated temperatures. For example, hydrogen as a reducing gas is oxidized to water, which then desorbs from the surface. The electrons released in this process pass into the conduction band as shown in figure 2.

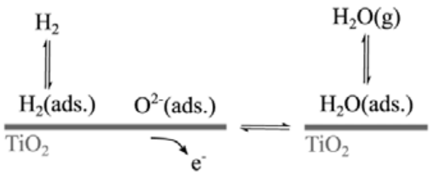

Figure 2. Reaction of hydrogen with adsorbed oxygen species [12].

Compared to the oxidized state, this will increase the charge carrier density in the conduction band resulting in a significant drop of electrical resistance. When the concentration of reducing gas decreases, more and more oxygen species are again adsorbed on the surface, hence reducing the charge carrier density and conductivity until the initial value is reached again. Vice versa, oxidizing gases are reduced on the sensor surface and decrease the conductivity i.e., increase the resistance. Thereby, the oxide layer can be used as a sensing device for oxidizing and reducing gases and vapors.

To quantify the properties of a gas sensor, the sensor response S_R is usually calculated by forming a ratio of the resistances measured in air and in the presence of a detectable gas. Equation 6 displays the calculation of sensor response S_R for reducing gases [19].

$$S_R = \frac{R_{air}}{R_{gas}} \qquad (6)$$

3. Experiments and Results

In this section, four experiments for upper secondary chemistry class and their results are presented: The first two experiments deal with the electrochemical synthesis of a nanoporous titanium dioxide layer, its characterization by means of SEM and the approximation of the obtained surface area. In the following experiment, the temperature-resistance-relationship of a semiconductor is examined using titanium dioxide as an example. The last experiment presents a titanium dioxide gas sensor used for the semi-quantitative detection of ethanol gas.

3.1. Electrochemical Synthesis of a Titanium Dioxide Layer

In this experiment, a nanoporous titanium dioxide layer is fabricated by anodic oxidation.

3.1.1. Experimental

Material: beaker, crocodile clamps, cables, power supply. heating plate, water bath, scissors, tweezers

Chemicals: titanium foil (thickness: 0.5 mm, e.g. from Fisher Scientific), ethylene glycol (GHS 07/08), ammonium fluoride (GHS 06), water, acetone (GHS 02/07)

The titanium foil is cut into pieces of roughly 4 cm x 2 cm, cleaned thoroughly with water and acetone and subsequently air-dried. Then, the titanium foil is anodized in an electrolyte made of ammonium fluoride (w = 0.5%) and water (w = 1%) in ethylene glycol for 45 min at 50 V DC. To keep the temperature at 40 °C the electrolyte is stirred at 600 rpm and placed in a water bath. After the anodization, the titanium foil is cleaned with water and acetone. Figure 3 shows the experimental setup.

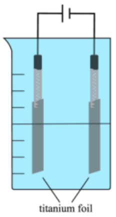

Figure 3. Experimental setup of the anodization. Titanium foil is used both as anode and cathode.

3.1.2. Results

During the process of anodization, a dull brown titanium dioxide layer is formed according to equation 2 and 3. Figure 4 shows the nanoporous titanium dioxide layer (referred to in the further course as "anodized titanium foil") in comparison to titanium metal.

Figure 4. Color of untreated titanium foil (left) and nanoporous titanium dioxide layer (right).

3.2. Characterization of Nanoporous Titanium Dioxide Layer by SEM and Calculation of Surface Area

The nanoporous titanium dioxide layer fabricated in 3.1 is examined with regards to the surface area. By a simple calculation, it can be shown that a nanoporous layer has a much higher surface area in comparison to a compact layer.

3.2.1. Experimental

We characterized the titanium dioxide layer using a scanning electron microscope (SEM) to show the nanoporous morphology and to determine pore sizes. Additionally, the side view of the porous layer provides an opportunity to measure the layer thickness, i.e., the length of the nanotubes. Subsequently, the surface area of a compact layer can be compared mathematically with that of a nanoporous surface.

3.2.2. Results

The SEM images reveal a nanoporous structure with nanotubes having an inner diameter d_{nanotube} of approx. 70 nm and a height h_{nanotube} of approx. 10 µm (Figure 5).

Using the values obtained in figure 5 and approximating the nanotubes as ideal cylinders, the shell surface of one nanotube (S_{nanotube}) can be calculated according to equation 7 which determines the shell surface. Figure 6 shows the approximation of titanium dioxide nanotubes as ideal cylinders.

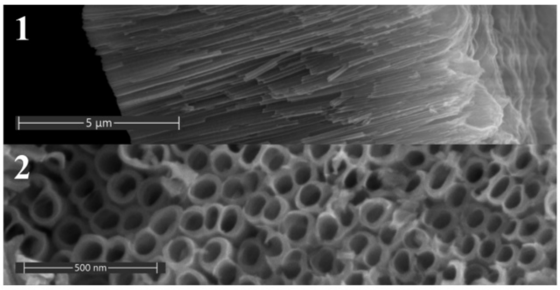

Figure 5. SEM images of nanoporous titanium dioxide layer (anodization time: 45 min) after heating to 350 °C (1: side view; 2: top view).

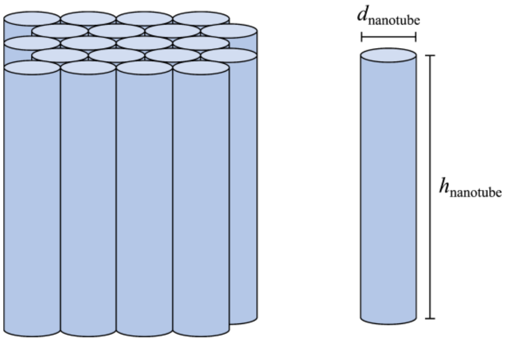

Figure 6. Model of the nanoporous titanium dioxide structure approximated as ideal cylinders.

$$S_{\text{nanotube}} = \pi \cdot d_{\text{nanotube}} \cdot h_{\text{nanotube}} \approx 2.2 * 10^{-12}\,\text{m}^2 \tag{7}$$

The SEM images reveal that about 90 nanotubes fit on a square surface of 1 µm^2. Hence, a number of 9 10^{13} nanotubes fits on a square surface of 1 m^2. Using these values, the surface area of all nanotubes on 1 m^2 can be calculated according to equation 8.

$$\begin{aligned} A_{\text{nanotube}} &= S_{\text{nanotube}} \cdot n_{\text{nanotube}} \\ &= 2.2 * 10^{-12}\,\text{m}^2 \cdot 9 * 10^{13} \\ &\approx 200\,\text{m}^2 \end{aligned} \tag{8}$$

Hence, the surface area of all nanotubes of a square surface of 1 m^2 is approx. 200 m^2, which indicates an area growth by a factor of approx. 200. Due to the larger surface area in comparison with a compact layer, nanoporous titanium dioxide is suitable for gas sensing purposes. This increase in surface area is in good agreement with scientific literature [20], determining a factor of approx. 100 for a much thinner oxide layer.

3.3. Temperature Dependence of the Resistance of a Semiconductor

In this experiment, the semiconductor property of the fabricated titanium dioxide is confirmed by the decreasing resistance at higher temperatures due to the higher number of electrons in the conduction band.

3.3.1. Experimental

Material: metal clamps (binder clips), crocodile clamps, cables, multimeter, ceramic heating plate, copper wire, tweezers, infrared thermometer

Chemicals: anodized titanium foil (see 3.1)

The experiment is set up according to figure 7.

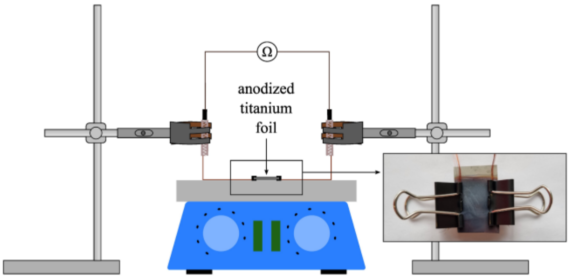

Figure 7. Experimental setup for experiment 3.3. The inset shows the attachment of copper wires to the oxide layer using binder clips.

The foil is carefully contacted by the two metal clamps on opposite sides connecting pieces of copper wire (approx. 5 cm) and placed in the middle of the plate. To exclude irreversible effects like carbonization of organic residues, crystallization or alterations in nano- and microstructure, the sample was initially heated to the highest temperature used during this experiment (i.e. 580 K). Afterwards, it was allowed to cool down to 130 °C (approx. 400 K) and the first resistance data point is collected. Afterwards, the temperature of the heating plate is increased in steps of 20 K. The corresponding sample temperature is measured using the infrared thermometer and plotted against the respective resistance. Suitable values can be obtained in the range from 400 to 580 K.

3.3.2. Results

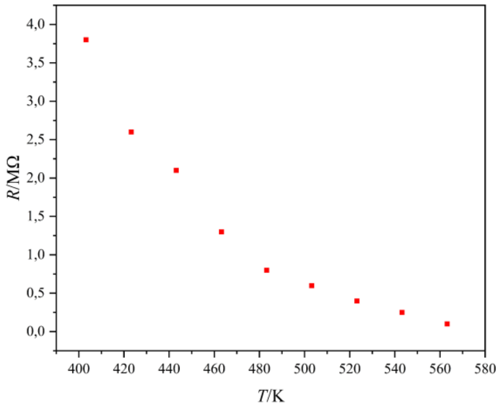

Figure 8. Electrical resistance R as a function of temperature T.

The electrical resistance drops when the temperature is increasing. The color of the surface turns from dull-brown into a bluish black. This may be attributed to a partial reduction of titanium dioxide [21]. Figure 8 shows exemplary measuring values.

The observed change in resistance in semiconducting materials can be explained using the band model: When the temperature increases, more electrons can be excited into the conduction band overcoming the band gap (figure 9) due to their higher energy, which leads to a higher conductivity and therefore a lower resistance.

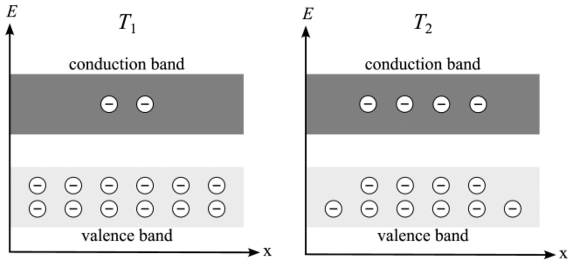

Figure 9. Band model of a semiconductor for two different temperatures T_1 and T_2 ($T_2 > T_1$).

3.4. Detection of Reducing Gases (semi-quantitative)

In this experiment, a semi-quantitative experiment presents the suitability of nanoporous titanium dioxide as a gas sensor. In order to keep a nearly constant atmosphere, the experiment is performed under a crystallizing dish or a large beaker. An aquarium air pump is used to provide a constant gas flow. As a reducing test substance, which is easy to handle and non-toxic, ethanol is used. Additionally, ethanol is a suitable model substance since semiconductor gas sensors are also used in police alcohol check instruments.

3.4.1. Experimental

Material: metal clamps (binder clips), crocodile clamps, cables, multimeter, ceramic heating plate, copper wire, crystallizing dish (or large beaker), tweezers, aquarium air pump, hose, glass tube (length approx. 2 cm), syringe

Chemicals: anodized titanium foil (see 3.1), ethanol (GHS 02/07)

The experiment is set up according to figure 10.

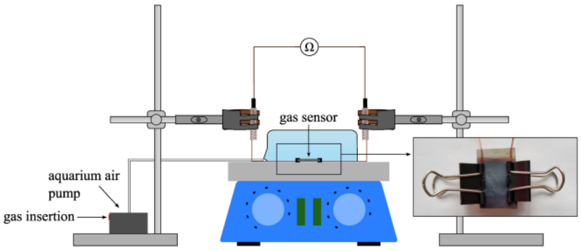

Figure 10. Experimental setup for experiment 3.4.

The foil is carefully contacted by the two metal clamps on opposite sides connecting pieces of copper wire (approx. 5 cm) and placed in the middle of the plate.

Subsequently, a crystallizing dish is placed upside down (as a chamber). The plate is heated to a temperature of 300 °C. The pump is set up to provide a constant air flow through the chamber. Then, ethanol vapor is diluted in the syringe to a ratio of 1:25. 5 mL, 10 mL and 20 mL, respectively, of the gas mixture are fed into the air pump. Meanwhile, the resistance is measured with the multimeter and digitally recorded. Afterwards, the experiment is repeated at a temperature of 350°C.

3.4.2. Results

Under an ambient air flow, the resistance of the sensor surface is stabilizing at an almost constant level. After inserting the gas mixture containing ethanol into the chamber, the electrical resistance drops rapidly and subsequently increases again until the initial value is approximately reached. Figure 11 shows the values measured for a working temperature of 300°C.

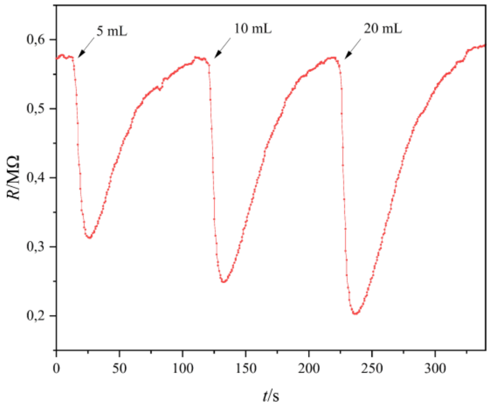

Figure 11. Electrical resistance *R* measured after the insertion of 5 mL, 10 mL and 20 mL ethanol, respectively (working temperature: 300°C).

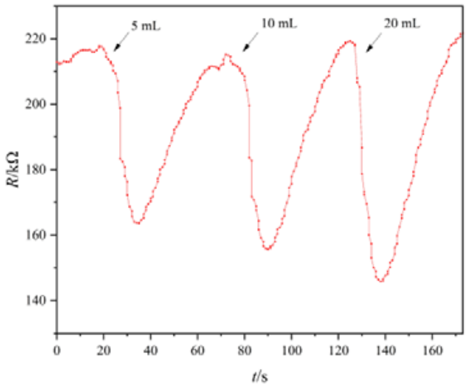

Figure 12. Electrical resistance *R* measured after the insertion of 5 mL, 10 mL and 20 mL ethanol, respectively (working temperature: 350 °C).

Figure 12 shows the values measured for a working temperature of 350°C.

Ethanol vapors have a reducing effect. Oxygen species which are adsorbed on the semiconducting titanium dioxide surface react with ethanol if the gas sensor is exposed to ethanol vapor. In [8], the reaction is formulated using O^- species for a $LaFeO_3$ gas sensor. As a didactical reduction, we suggest to assume the presence of O^{2-} ions which are more familiar to students as oxide ions (equation 9).

$$C_2H_6O + 6\ O^{2-}(ads.) \rightarrow 2\ CO_2 + 3\ H_2O + 12\ e^- \quad (9)$$

Hence, electrons are released into the conduction band; the electrical resistance drops (sensor response). Due to the high temperature of the sensor, reaction products desorb from the surface and new air oxygen can be adsorbed (recovery).

To quantify the sensor response, the relative sensor response S_R can be calculated exemplarily according to equation 6. Therefore, values of R_{air} and R_{gas} are read from figure 11 and 12. Table 1 displays the values gained from figure 11 and 12.

Table 1. Sensor Responses S_R Calculated for a Working Temperature of 300 °C and 350 °C and Different Gas Amounts, respectively.

T = 300 °C				T = 350 °C			
V_{test}/mL	5	10	20	V_{test}/mL	5	10	20
R_{air}/MΩ	0.58	0.57	0.57	R_{air}/kΩ	215	212	219
R_{gas}/MΩ	0.31	0.25	0.21	R_{gas}/kΩ	163	155	146
S_R	1.87	2.28	2.71	S_R	1.32	1.37	1.5

The sensor response S_R hence depends on the amount of ethanol gas to which the sensor is exposed. The more ethanol is presented to the sensor, the higher the sensor response is. Moreover, a higher working temperature does not necessarily lead to a higher sensor response, which matches the results of [22] where a maximum sensitivity for a certain gas sensor at a specific temperature was determined.

4. Discussion and Educational Aspects

The construction of a gas sensor based on a simple titanium foil offers a wide range of learning opportunities in upper-secondary chemistry class. In the first step, the anodic oxidation process can be used to repeat and apply numerous fundamentals from the field of electrochemistry. This includes, for example, the construction and operation principle of an electrolytic cell and the chemical processes at the electrodes and in the electrolyte.

Consequently, temperature-dependent resistance measurements of the nanoporous oxide layer can be used to introduce the semiconducting properties of titanium dioxide in class by simple setup. It can be demonstrated that even small changes in the environment, such as fanning air or breathing on the metal oxide surface, have a significant effect on its surface resistance. Similar effects cannot be observed with metallic and non-porous oxidic samples of e.g. titanium, copper, iron or similar. A simple band model provides a suitable didactically reduced description of the phenomenon, whereby the role of oxygen defects in the TiO_2 lattice as electron donors can also be taken up in advanced learning groups. After establishing the connection between the influence of the environment and surface resistance, the question of the extent to which the composition of the ambient atmosphere influences the surface resistance of nanoporous titanium dioxide can be investigated. Simple hands-on experiments, such as the careful application of ethanol vapor from a squirt bottle, shows that this significantly reduces the surface resistance, whereas the gas supply of oxygen leads to a slight increase in the electrical resistance.

The interpretation of the phenomena can be understood using the ionosorption model which refers to basic chemical concepts like the donor-acceptor concept as well as the energy concept. Depending on the time frame, low-cost experimental setups can be realized in further student projects, with which semi-quantitative gas concentrations can be determined (see 3.4). In this setting, students can develop their own research questions, practice the use of digital data acquisition, implement the graphical representation of measured values and apply mathematical tools and analysis methods, e.g. for determining the relative sensor response. Furthermore, cooperation with a university operating a scanning electron microscope (SEM) can enable the students to characterize their own oxide layers to increase motivation and engagement.

Acknowledgement

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