

CHEMICAL SENSORS

Properties, Performance and Applications

*Chemistry Research and
Applications Series*

Ronald V. Harrison
Editor

NOVA

CHEMISTRY RESEARCH AND APPLICATIONS

CHEMICAL SENSORS: PROPERTIES, PERFORMANCE AND APPLICATIONS

RONALD V. HARRISON

EDITOR



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PREFACE

Ozone is a harmful gas to people's health even at low concentrations. Thus, it has attracted much interest to develop portable energy-saving high-resolution ozone sensors. In this book, the physical principle of photon stimulated ozone sensors based on indium oxide nanostructures has been investigated. Ozone sensors have been integrated with light-emitting diodes (LEDs) and the sensor performance towards real applications has been tested. To examine the mechanisms of photon stimulation (photoreduction) and oxidation effects, electrical, surface analytical and structural characterization of ozone sensing layers were performed and analyzed. Moreover, optical fiber sensor has found applications in the biomedical research, industrial process control and environmental monitoring. This book provides a review of the optical fiber sensor, especially focused on the optical fiber sensor development and its application in gas detection. In addition, electrochemical impedance spectroscopy (EIS) is a sensitive tool providing information on various physical and chemical properties of materials, as well as on interaction processes occurring in the bulk or at the surface of these materials. In this book, the use of impedimetric transducers based on interdigitated electrode arrays (IDEA) for chemical and bio-sensors development is reviewed. Different designs of IDEA devices are presented and the effect of the transducer geometry on resulting impedance spectra is discussed. The authors also examine the development of an amperometric biosensor for phenol detection. The variables that exert influence on the performance of the biosensor response, including enzyme immobilization procedure, laccase amounts, pH and working potential were investigated as well. Furthermore, the feasibility of the biosensor response for various phenol compounds was also investigated. Recent advances in sensor technology, signal processing and pattern recognition algorithms have led to the development of chemical sensing instruments housing one or more non-specific gas sensors. This book also reviews the recent applications of non-specific gas sensor array technologies used for environmental monitoring of odours; including a brief history on odour measurement applications; the different types of sensors utilised in gas sensor array systems and a range of pattern recognition techniques, from simple statistical analyses to artificial neural networks, used for the purpose of odour identification and quantification is also discussed.

Chapter 1 - Ozone is a harmful gas to people's health even at low concentrations (< 100 parts per billion, ppb). Thus, it has attracted much interest to develop portable energy-saving high-resolution ozone sensors. Photon stimulated gas sensors operated at room temperature are very suitable for such applications. In this work, the physical principle of photon stimulated ozone sensors based on indium oxide nanostructures has been investigated. Ozone

sensors have been integrated with light-emitting diodes (LEDs) and the sensor performance towards real applications has been tested. To examine the mechanisms of photon stimulation (photoreduction) and oxidation effects, electrical, surface analytical and structural characterization of ozone sensing layers were performed. The adsorbed oxygen species, which was analyzed to be O^- compared to the widely accepted O_3^- in the literature, plays a major role during ozone sensing, and the adsorption and desorption process takes place mainly at the nanoparticle surfaces. Furthermore, the photon-stimulation process occurs throughout the sensing layer, while gas diffusion dominates in the oxidation process. A model describing ozone sensors based on high-deficiency indium oxide nanoparticles is proposed.

Based on the physical principles of photon stimulation and O_3 oxidation, ozone sensors integrated with ultraviolet LEDs were fabricated. First, different operation modes (pulse or continuous mode) were tested, and the ideal operation temperature of ozone sensors was determined. The required photon energy and light intensity were also determined for the reactivation of the active In_2O_3 layer. The ozone response (defined as a ratio between the layer resistances after O_3 oxidation and photoreduction) was determined to be greater than 10^5 in vacuum. In synthesized air, the sensor also showed good ozone sensing results. The lowest detected ozone concentration was found to be as low as ~ 13 ppb. In addition, this type of ozone sensor showed a good long-term stability and a low cross-sensitivity against other oxidizing gases, such as NO_x , CO_2 , O_2 . Furthermore, measurements under real conditions were carried out by an ozone sensor demonstrator. Ozone with a concentration as low as 12 ppb was detected in real atmosphere. The energy consumption of such sensors including electrical circuit was determined to be lower than 50 mW. Thus, compact, portable, low-cost, environmental ozone sensors were developed, which can operate at room temperature and are suitable for further integration in plastic packages.

Chapter 2 - Optical fiber sensor technology has experienced significant growth since its early beginning in the 1970s and becomes more sophisticated since mid 1990s. Optical fiber sensors have found applications in biomedical research, industrial process control and environmental monitoring. This chapter provides a review of the optical fiber sensors, especially focused on the optical fiber sensor development and their applications in gas detection.

Chapter 3 - Electrochemical impedance spectroscopy (EIS) is a sensitive tool providing information on various physical and chemical properties of materials, as well as on interaction processes occurring in the bulk or at the surface of these materials. In the present chapter, the use of impedimetric transducers based on interdigitated electrode arrays (IDEA) for chemical and bio-sensors development is reviewed. Examples of IDEA applications as ion sensors, immunosensors, DNA sensors and enzyme biosensors are given. Different designs of IDEA devices are presented, and the effect of the transducer geometry on resulting impedance spectra is discussed in terms of the electrical equivalent circuit components. Technological aspects of sensor fabrication are concerned and, finally, a new design of a three-dimensional IDEA impedimetric sensor is presented along with its fabrication technology. Due to the presence of insulating barriers that separate the adjacent digits of the electrodes, the main portion of the probing electrical current goes close to the surface of the barrier. Chemical modification of the barrier surface with biomolecules (antigens, DNA, enzymes) permits the realization of direct label-free detection of subsequent analytes in solution with high sensitivity.

Chapter 4 - Fluorescence is nowadays the preferred photoluminescence detection mode in the development of novel optical sensors, mainly due to the large amount of fluorescent indicators available for a wide variety of different analytes. However, room temperature phosphorescence (RTP) is gaining importance and popularity as an alternative highly selective detection mode for luminescent optical sensing. In fact, an increased number of RTP publications have appeared in the last years dealing with the successful implementation of new measurement methods and new solid supports for the development of such sensing materials. Moreover, energy transfer-based sensing schemes offer a promising approach to overcome the lack of available phosphorescent indicators (one of the main limitations hindering the development of phosphorescence-based optosensors).

The coupling of highly selective recognition elements, such as molecular imprinted polymers, followed by a phosphorescent detection has proved to be a powerful tool for the development of highly sensitive and selective optosensors based on phosphorescence detection.

A particular field where phosphorescent sensing is playing an increasingly important role is the development of “in vivo” sensing applications. Oxygen sensing based on the quenching of phosphorescence has genuine potential for clinical applications, such as cerebrovascular and cardiovascular events and the detection of tumors. Lately, phosphorescence based sensing is achieving greater applicability and so new and more robust “in vivo” RTP optical sensors are expected in the near future.

Chapter 5 - Recent advances in sensor technology, signal processing and pattern recognition algorithms have led to the development of chemical sensing instruments housing one or more non-specific gas sensors. These instruments are often referred to as ‘electronic noses’ or ‘artificial olfaction systems’ since they measure a range of non-specific compounds that make up odorous emissions. Odours usually contain various odorous chemical compounds, some of which cause annoyance to local receptors. For this reason, odour measurement has often been considered more of an art than a science. Subjective sensory measurement using a human nose has been the accepted ‘standard’ for odour measurement. This chapter reviews the recent applications of non-specific gas sensor array technologies used for environmental monitoring of odours; including a brief history on odour measurement applications; the different types of sensors utilised in gas sensor array systems are categorised and reviewed according to their operating principles; a range of pattern recognition techniques, from simple statistical analyses to artificial neural networks, used for the purpose of odour identification and quantification is also discussed. Considerable work has already been carried out in a broad range of industries including agriculture, food, waste treatment, paper, and tannery operations. In addition, there are efforts to develop on-site or portable sensor array devices for real-time and continuous odour measurement, capitalising on the low-cost, non-invasive, operator-free advantages of non-specific chemical gas sensor array technology.

Chapter 6 - In the present work is reported the development of an amperometric biosensor for phenol detection based in the coimmobilization of the Meldola’s Blue, substituting the role of the molecular oxygen in the regeneration of the enzyme and laccase via EDC on oxidized multi-wall carbon nanotubes (MWCT_{oxi}). The variables that exert influence on the performance of the biosensor response, including enzyme immobilization procedure, laccase amounts, pH and working potential were investigated. Furthermore, the feasibility of the biosensor response for various phenol compounds was also investigated. The

amperometric response for catechol using the proposed biosensor showed a wide linear response range (1 to 150 $\mu\text{mol L}^{-1}$), good sensitivity ($57.1 \mu\text{A cm}^{-2} \mu\text{mol}^{-1} \text{L}$), excellent operational stability (after 200 determinations the response remained at 97 %) and very good storage stability (lifetime > 3 months). The results were compared with laccase immobilized on graphite powder, highlighting the remarkable features of MWCT_{oxi} in the biosensor performance. According to these features, it is possible to affirm that the developed biosensor is promising tool for phenol detection due to its good electrochemical response and enzyme stabilization.

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Chapter 1

PHOTON STIMULATED OZONE SENSING

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ABSTRACT

Ozone is a harmful gas to people's health even at low concentrations (< 100 parts per billion, ppb). Thus, it has attracted much interest to develop portable energy-saving high-resolution ozone sensors. Photon stimulated gas sensors operated at room temperature are very suitable for such applications. In this work, the physical principle of photon stimulated ozone sensors based on indium oxide nanostructures has been investigated. Ozone sensors have been integrated with light-emitting diodes (LEDs) and the sensor performance towards real applications has been tested. To examine the mechanisms of photon stimulation (photoreduction) and oxidation effects, electrical, surface analytical and structural characterization of ozone sensing layers were performed. The adsorbed oxygen species, which was analyzed to be O^- compared to the widely accepted O_3^- in the literature, plays a major role during ozone sensing, and the adsorption and desorption process takes place mainly at the nanoparticle surfaces. Furthermore, the photon-stimulation process occurs throughout the sensing layer, while gas diffusion dominates in the oxidation process. A model describing ozone sensors based on high-deficiency indium oxide nanoparticles is proposed.

Based on the physical principles of photon stimulation and O_3 oxidation, ozone sensors integrated with ultraviolet LEDs were fabricated. First, different operation modes (pulse or continuous mode) were tested, and the ideal operation temperature of ozone sensors was determined. The required photon energy and light intensity were also determined for the reactivation of the active In_2O_3 layer. The ozone response (defined as a ratio between the layer resistances after O_3 oxidation and photoreduction) was determined to be greater than 10^5 in vacuum. In synthesized air, the sensor also showed

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good ozone sensing results. The lowest detected ozone concentration was found to be as low as ~ 13 ppb. In addition, this type of ozone sensor showed a good long-term stability and a low cross-sensitivity against other oxidizing gases, such as NO_x , CO_2 , O_2 . Furthermore, measurements under real conditions were carried out by an ozone sensor demonstrator. Ozone with a concentration as low as 12 ppb was detected in real atmosphere. The energy consumption of such sensors including electrical circuit was determined to be lower than 50 mW. Thus, compact, portable, low-cost, environmental ozone sensors were developed, which can operate at room temperature and are suitable for further integration in plastic packages.

1. INTRODUCTION

1.1 Necessities of Ozone Monitoring

As is well known, there exists an ozone layer in the earth's atmosphere, which is mainly located in the lower portion of the stratosphere at approximately 15 to 35 km above earth's surface. Ozone absorbs light in the ultraviolet (UV) spectral region of the sun between 200 and 320 nm, with an absorption peak located a 254 nm. The sun's radiation between 200 and 290 nm is most damaging to DNA molecules. [1, 2] Stratospheric ozone prevents highly energetic radiation from reaching the earth's surface, providing protection of all terrestrial life. Furthermore, ozone is a powerful oxidizing agent. Due to its high oxidizing potential, ozone is applied for sterilization in fish farms and in hospital operating rooms, disinfection of industrial and chemical freshwater, process water, and cooling water as well as disinfection, detoxification, deodorization of industrial wastewater. [3] For example, 95% of potable water in western Europe is treated by ozone.



Figure 1.1. A pollution measuring station in the center of Freiburg city in Germany.

However, as a negative effect of the powerful oxidizing potential, high concentration ozone gas can harm people's health, although ozone is often undesirably generated in everyday life, e.g. by older generation photocopiers and laser printers. Exposure of 0.1 to 1

ppm (parts per million) causes headache, burning eyes, and irritation to the respiratory passages. An individual remaining in a 0.1 ppm O_3 environment for two hours will sustain a loss of 20% in breathing capacity, and after remaining in 1 ppm O_3 for six hours, will suffer from an attack of bronchitis. A mouse kept in 10 ppm O_3 will not survive. [4] To protect people against ozone, threshold values of ozone concentration have been defined by European Guidelines (2002/3/EG). [5] The maximum permissible concentration of ozone is $240 \mu\text{g}/\text{m}^3$ (~ 120 ppb, parts per billion). A warning should be given at an ozone concentration of $180 \mu\text{g}/\text{m}^3$ (~ 90 ppb). The permissible highest 8-hour mean value of ozone concentration on any day will be reduced to $120 \mu\text{g}/\text{m}^3$ (~ 60 ppb) in 2010. Furthermore, the maximum ozone concentration in workplaces should not exceed $200 \mu\text{g}/\text{m}^3$ (~ 100 ppb). According to the air quality standard established by the U.S. environment protection agency, the standard for maximum ozone concentration for hourly exposure is set to be lower than ~ 80 ppb. [6] Thus, it is of great importance and interest to monitor ozone concentration in the low ppb range, i. e. lower than 60 ppb. That means the developed environmental ozone sensor must be able to detect less than 60 ozone molecules in 10^9 other molecules in its environment.

It is worth noting that ozone is unstable and is converted to oxygen with a half-life of about one hour at room temperature. [7] Furthermore, it can react easily with other atoms and donate a free oxygen atom to nitrogen, hydrogen, or chlorine. Thus, ozone concentration varies strongly with time and location. It is therefore very important to control environmental O_3 concentration both in workplaces, preventing O_3 leaks from O_3 generators, and in our everyday life, monitoring O_3 concentration for people's health. As a result, the monitoring of O_3 concentration becomes more and more requested. Alone in Baden-Württemberg, Germany, there are over 40 pollution measuring stations, monitoring exhaust gases such as O_3 and NO_2 . Ozone concentrations of more than $200 \mu\text{g}/\text{m}^3$ (~ 100 ppb) or even higher than the maximal permissible concentration of $240 \mu\text{g}/\text{m}^3$ (~ 120 ppb) has often been measured in past summers. The maximum value of ozone concentration was determined to be $282 \mu\text{g}/\text{m}^3$ (141 ppb) in Schwartenberg in Germany in 2007, which was much higher than the legal limit. [8] In Figure 1.1, an ozone measuring station in the center of Freiburg city is shown, indicating a 1-hour mean value of ozone concentration of $110 \mu\text{g}/\text{m}^3$ (~ 55 ppb). Such ozone measuring stations are very large and expensive. It would be impossible to install hundreds or even thousands of such measuring stations in one city to monitor the ozone concentration anytime and anywhere. However, ozone monitoring is an urgent demand. An alarm should be given at a high ozone concentration by portable devices, e. g. for sportsman exercising outdoors, or for children playing outside the nursery. Thus, portable low-cost and energy-saving ozone sensors are of great interest.

In this work, the development of a new type of small-size, portable, low-cost and energy-saving ozone sensor is described. In contrast to many efforts which have already been performed to develop ozone sensors based on different principles, such as the UV absorption method [9] or the electrochemical principle [10], an ozone sensor using semiconductor materials based on photon stimulation is developed. To compare the advantages and disadvantages of ozone sensor types, different sensing principles will be discussed in the following chapter. The new principle of photon stimulation for ozone sensors will also be described. For ozone sensing, indium oxide (In_2O_3), an n-type semiconductor material, is chosen due to its unique optical properties. Because the structural, electrical and optical properties of the material are very important for the sensor performance, an introduction of the material system will be given. After that, the growth of In_2O_3 nanostructures, acting as