

Enhancing the Potential of MOX-Based Gas Sensor Through iCVD Coatings for Biomedical Applications [†]

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Abstract

Nowadays, the medical sector challenges young research teams to develop and propose new non-invasive diagnostic methods. As a potential response, gas sensors for biomarker detection in exhaled breath show promising results. In this paper, various gas sensors based on metal–oxide semiconductors and coated with different polymers are proposed, demonstrating the potential of these sensors in breathomics and health breath tests. The proposed sensors are based on TiO₂ sensing structures and are tuned through different methods. Furthermore, they are coated with polymers such as PV4D4, PTFE, PV3D3, and copolymers such as P(V3D3 + TFE). These polymers show improved efficiency for gas sensing structures as they act as filters for certain molecules.

Keywords: PV4D4; PTFE; copolymer; P(V3D3 + TFE); TiO₂; hydrogen; breathomics; metal oxide; 2-propanol

1. Introduction

While technological progress has advanced considerably in the medical field compared to previous millennia, many causes of mortality persist. These include various cancers, respiratory diseases, infections, and various chronic diseases [1–4]. One of the major challenges currently facing research groups is the development of new, efficient, affordable, and precise methods for the detection of these diseases. To respond to this demand, this study focuses on breathomics, which offers various breath prints and valuable information on biomarkers in exhaled breath.

The amount of information one can detect from exhaled breath varies from source to source; however, it is reported to range from more than 1400 components [5] up to 3500 components [6]. These compounds are present in concentrations ranging from parts per million (ppm) to parts per billion (ppb), with some analytes reaching concentrations as low as parts per trillion (ppt) [7]. While the number and concentrations of these compounds are well known thanks to advanced analytical technologies, the use of breathomics can be traced back to ancient Greek medicine, where physicians identified diseases such as liver cirrhosis and diabetes through distinctive breath odors [1].

Thus, certain diseases, through various metabolic processes, result in the production of diverse analytes that are exhaled via blood–gas exchange in the lungs. Furthermore, many of these biomarkers have been identified and reported in the literature. While the detection of targeted gases and volatile organic compounds (VOCs) is the main focus of disease



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diagnostics, the importance of feedback systems, detectors, and sensors for monitoring and controlling medical gases used for therapeutic purposes should not be overlooked [8–11].

Some of the analytes of interest in exhaled breath currently under investigation include ammonia, acetone, hydrogen, carbon dioxide, 2-propanol, n-butanol, and ethanol, while our research group is also investigating solutions for formaldehyde and methane. Hydrogen works as both a therapeutic gas [12–14] and a biomarker for various gastric diseases [15–17]. Ammonia is usually associated with the liver [18–20] and kidney [21,22] diseases, as well as being the product of *Helicobacter pylori* [19,23]; 2-propanol and n-butanol are potentially linked to various cancers [24–28]. Ethanol is linked to alcohol levels in the blood [29–31] and has recently been associated with auto-brewery syndrome [32,33]; and acetone is a glycemic biomarker [28,34]. On the other hand, while carbon dioxide is not necessarily used as a biomarker, its detection is used in capnometry [35,36], as well as in the medical field, in various operations.

In this paper, some solutions are proposed based on various gas sensors coated with different polymeric structures, such as PV4D4 with as-grown [37] and annealed structures [35,38], as well as coated with Teflon (PTFE) [39] and the copolymer combination P(V3D3 + TFE) [40] for achieving results in biomarker detection. While the research pool and many papers offer their solutions, we propose novel composite nanomaterials, as relatively new manufacturing methods are used for the fabrication of these sensors. Through iCVD, thin films of polymers are obtained with a high-precision control over the coating thickness [41,42].

2. Gas Sensor Solutions

2.1. Gas Sensors

One particular approach in the field of gas sensors is to fabricate them using metal-oxide semiconductors. While there are multiple methods for fabricating gas sensors, the choice usually depends on the materials intended for use. For TiO_2 , commonly used techniques include spray pyrolysis [43], sputtering [44] and atomic layer deposition [45]. The polymer coatings were made via the iCVD reactor mentioned in previous papers [35,37–40,42,43,46], with careful attention paid to the deposition parameters for each polymer. Because they are dielectric, coatings produced via the iCVD usually exhibit “filter” properties. They are therefore tolerant to water vapor and increase selectivity toward specific analytes. Thus, using such coatings, various sensors have previously shown good repeatability with errors of up to 10% for gas response and long-term stability in terms of selectivity and sensitivity [8–11,35,37–40,42,43,47,48].

2.2. Improvement by Coatings

Figure 1 presents a comparison of various coatings for gas sensors, recently investigated for hydrogen detection at a concentration of 100 ppm.

In Figure 1, it can be observed that the greatest improvement in H_2 detection is obtained by applying the PV4D4 cage-structured polymer, followed by the transition structure consisting of PV4D4 rings transitioning to cages. Considering that MOX-based sensors usually operate at relatively high temperatures, the best responses were registered at 300 and 350 °C, and these responses are further improved by the polymer coating. Thus, PV4D4 annealed to a cage structure showed responses of 573% and 709%, respectively, while PV4D4 annealed to a transitioning stage between rings and cages exhibited the best responses of 175% and 230%, respectively.

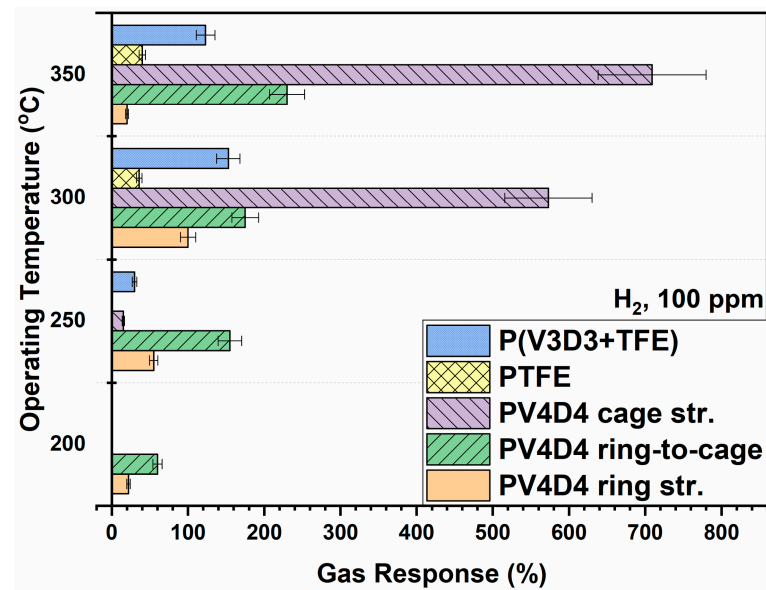


Figure 1. Comparison graph on five samples based on tuned TiO₂ gas sensing structures, coated with different polymer structure and their improvement on H₂ detection at 100 ppm concentration.

Figure 2 presents a comparison diagram of various coatings on gas sensors, as shown in Figure 1, recently investigated for 2-propanol detection at 100 ppm.

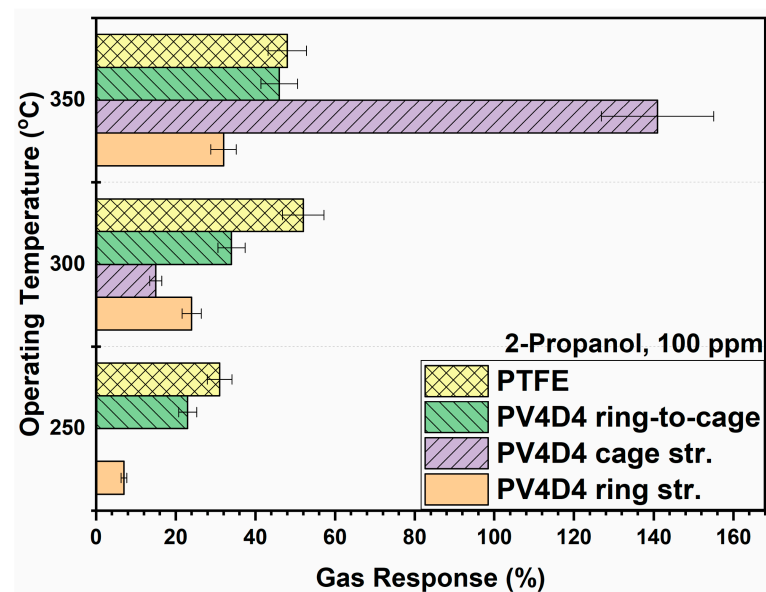


Figure 2. Comparison graph on four samples based on tuned TiO₂ gas sensing structures, coated with different polymer structure and their improvement on 2-propanol detection at 100 ppm concentration.

In Figure 2, it can be observed that the comparison of the discussed samples has been reduced to four. It is necessary to mention that the copolymer structure P(V3D3 + TFE) did not show a response to 2-propanol; therefore, it was deemed unnecessary to be included in the graph. Furthermore, the same tendency can be observed, as the best response was registered at an operating temperature of 350 °C for the PV4D4 cage structure, followed by the PTFE coating. Thus, the best response for the PV4D4 cage structure was 141% at 350 °C, while the next-best response was 46% for the PTFE coating at 300 °C.

3. Discussion

While this paper presents only a small part of the research, several important observations must be mentioned. Firstly, polymer coatings are commonly used for their hydrophobic properties. As they are typically dielectric, their various structures enable them to repel water vapor effectively. Thus, to better understand the effect of the polymers used, additional research on their hydrophobic properties is required. On the other hand, thin-film polymers show high potential as filters, thereby increasing selectivity toward different targeted analytes, as can be observed in the present study. Although a wide range of gases used in the medical field were investigated, the focus of this paper was placed on hydrogen and 2-propanol sensors due to their emerging importance and the significant attention they have attracted.

If we were to map the development of gas sensors, we would observe a complex interplay of materials, many of which yield unique results owing to the variability of composite structures. New technologies are being developed daily to improve, tune, and optimize known base materials for gas sensors, while simultaneously attempting to minimize the influence of various interfering factors. To highlight the novelty of the proposed work, Table 1 provides a comparison of selected hydrogen sensors developed for different applications.

Table 1. Comparison table of H₂ responses for various tuned gas sensors.

Sensor Base	Nanoparticles	Concentration, ppm	Response (%)	OPT (°C)	Response/Recovery (s)	REF
ZnO + 2% Ag	Ag	250	4357	350	—	[49]
ZnO + 1 at% Ag	Ag	100	2200	300	6.6/—	[50]
TiO ₂ nanotuburi + ZnO	ZnO	100	520	350	22/>2000	[51]
TiO ₂ + AgAu NPs	AgAu	100	300	350	0.7/1.5	[35]
TiO ₂ + Pt NPs	Pt + UV	1% (10,000 ppm)	240	Room Temperature (RT)	—	[52]
SnO ₂ nanofibre + Pd	Pd	1000	224	300	8.4/—	[53]
Pd/SiO ₂ @TiO ₂	Pd + core-shell	1000	2283 (S = 22.83)	25 (RT)	68/43	[54]
WO ₃ + PtO _x /PdO _γ	Co PtO _x + PdO _γ	100	~200 (estimated)	110	—	[55]
TiO ₂ aerogel 3D + Pd	Pd	100	480 (S = 4.8)	325	<2/<22	[56]

As can be observed, there are already many studies in which the improvement is limited to using various nanoparticles, which certainly offer the desired effect, yet rely on a range of different materials. However, by improving some sensors with polymer coatings, a second function could be achieved for the same sensors. This improvement was already demonstrated in previous papers, in which two-in-one sensors were obtained for two different biomarkers by applying a PV4D4 coating to TiO₂ gas sensors. In one case, 100 ppm of ammonia was detectable at room temperature, while 100 ppm of hydrogen was detected at relatively higher operating temperatures (up to 350 °C). In the second case, hydrogen exhibited an n-type response from 150 to 350 °C, while carbon dioxide exhibited a p-type response from 150 to 250 °C, enabling the sensor to detect two analytes simultaneously. This was possible because the PV4D4 polymer was used in two different structural phases: as-grown and thermally annealed to a transitioning ring-to-cage structure. The nearly full cage structure showed significantly improved selectivity toward H₂, as well as high sensitivity. Therefore, a second table is proposed. In Table 2, it can be observed that, for similar structures, applying polymer coatings provides better responses to 2-propanol.

Table 2. Comparison table of 2-propanol responses for various tuned gas sensors.

Sensor Base	Polymer	Concentration, ppm	Response	OPT (C)	REF
SnO ₂	-	40	20.1 Ra/Rg-1	75	[57]
ZnO/CdO	-	50	10.23 Ra/Rg	248	[58]
TiO ₂	-	200	1.68 Ra/Rg	300	[59]
CuO	-	100	4.66 Ra/Rg	200	[60]
SnO ₂ /ZnO	-	500	1.99 Ra/Rg	300	[61]
ZnSnO ₃	-	10	10.3 Ra/Rg	200	[62]
NiFe ₂ O ₄	-	20	6.51 Ra/Rg	120	[63]
TiO ₂	PV4D4 (as grown)	100	251%	350	[42]
TiO ₂	PV4D4 (as grown)	100	225%	400	[42]
TiO ₂	P(V3D3 + TFE)	100	64%	350	[40]
CuO	PV4D4	100	~64%	300	[42]
TiO ₂	PV4D4 (as grown)	100	251%	350	[42]

As can be observed, polymer coatings tend to increase the response value in some cases when used with other materials. However, the use of hydrophobic polymers such as Teflon (PTFE) and PV4D4 already offers significant improvement and enables their application in medical research. In contrast, many conventional metal–oxide-based gas sensors are limited in humid environments unless they are specifically designed as humidity sensors.

The improvement and tuning of gas sensor properties are constantly adapting to new technologies. Thus, using initiated chemical vapor deposition (iCVD), it becomes possible to coat sensors with high precision and to select the optimal polymer thin-film structure for specific applications. Furthermore, some polymers, such as PV4D4, can change their structure through thermal annealing, thereby offering a relatively simple and versatile method for further tuning gas sensor performance.

4. Conclusions

This paper proposes alternative solutions for the gas sensor industry that are worthy of further research. These solutions focus on improving and adapting methods to tune the studied gas sensing structures for specific tasks, such as non-invasive diagnostics in the medical field and precise feedback control in gas delivery systems.

From the graphs selected and presented in this paper, it can be concluded that in specific areas where hydrogen and 2-propanol need to be monitored, TiO₂-based gas sensors coated with either annealed PV4D4 or PTFE can be effectively used. As mentioned earlier, thermal treatments allow polymers to change their structure, thereby offering versatile properties tailored to targeted needs.

In conclusion, further research is still needed to determine which combinations of polymers and gas sensing structures perform best for various specific industrial and medical applications.

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