

Broadening ZnO: Ag Potential for Hydrogen Detection Applications via iCVD-Coated Thin-Film Polymer [†]

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Abstract

Considering the various applications where detection is required—whether for industrial or medical purposes—providing cost-efficient, stable, and highly selective solutions remains challenging. Different industries require hydrogen detectors, because this gas has the potential to serve as a clean and versatile energy carrier; therefore, precise monitoring and feedback in such systems are of enormous importance. Simultaneously, the medical field is developing new therapeutic methods using hydrogen as a medical gas, while also utilizing it as a biomarker in exhaled breath for various gastric diseases. In this paper, a ZnO-based gas sensor, doped with Ag nanoparticles produced via the Solution Chemical Synthesis (SCS) method, was coated with a thin polymer film of poly(1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetrasiloxane) PV4D4 via initiated Chemical Vapor Deposition (iCVD). The results are promising: at a relatively high operating temperature of 350 °C, the sensor showed its highest registered response to H₂ gas (up to 23%). Compared to other gases studied at the same temperature, the sensor also showed potential for detecting 2-propanol, n-butanol, and ethanol, albeit with lower responses. Based on the dynamic response analysis, the fastest reaction time was also recorded at the highest operating temperature, thus showing versatile possibilities for using the specified detector.

Keywords: zinc oxide; PV4D4; gas sensors; silver; nanoparticles



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1. Introduction

Hydrogen, recognized as an environmental component used in various industries, requires monitoring across multiple fields, including oil-immersed transformers, household, hospital, and energy-related niches [1–5]. It has gained vital importance due to its significant applications, such as its use as a therapeutic and preventive gas [6–11], in energy applications and fuel cells [7,12–14], and in hydrogen-powered vehicles [15–17]. However, if hydrogen concentrations exceed 4% in the air, there is a risk of explosion [18–20]; therefore, battery rooms, for example, should be ventilated to avoid such risks [2].

As straightforward as it may seem, gas detectors are required to address these specific needs. There is significant interest in this field regarding gas sensors based on metal oxides

that are tuned through various technologies. Previous research [21–23] has shown that ZnO has the potential to be used as such a solution. Hydrogen sensors are obtained through various methods and materials; for instance, the hydrothermal method is used to synthesize ZnO nanowires [24]. The authors demonstrated that different types of treatments allowed them to obtain ZnO nanowires with high performance in detecting various hydrogen concentrations. Another approach to developing hydrogen sensors involves the use of noble metals such as Ag and AgPt [25]. Furthermore, functionalization with Pd nanodots has enabled the development of hydrogen sensors based on copper oxide [25] and ZnO sensors doped with Eu impurities [26]. Additionally, an Al₂O₃/CuO-based sensor allowed for hydrogen detection even under high humidity conditions. In that case, copper oxide was initially obtained by the Solution Chemical Synthesis (SCS) method, followed by the deposition of aluminum oxide using Atomic Layer Deposition (ALD) [27,28]. Similarly, a selective sensor for hydrogen detection was demonstrated based on copper oxide doped with Ni impurities [29].

Recently, the use of different types of polymers has become particularly important for the development of various gas sensors. For instance, the use of poly(1,3,5-Trivinyl-1,3,5-trimethylcyclotrisiloxane) PV3D3 deposited over CuO/Cu₂O/ZnO:Fe structures has made it possible to obtain sensors that are stable at high humidity levels when detecting hydrogen molecules. In their paper, the authors reported that while CuO/Cu₂O/ZnO:Fe was initially selective to ethanol molecules, after depositing the PV3D3 polymer, the selectivity shifted toward hydrogen [30]. The poly(1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetrasiloxane) PV4D4 deposited on TiO₂ structures treated at different temperatures allowed the development of sensors selective to hydrogen molecules [31,32]. By combining mixed oxides of CuO and ZnO, a sensor selective to hydrogen was obtained [2].

Another type of hydrogen detection sensor is based on MOF structures deposited on copper oxide and doped with Al impurities [33–35]. In these papers, the authors demonstrated the development of selective sensors capable of detecting different hydrogen concentrations under various relative humidity levels. This paper presents a hydrogen sensor based on zinc oxide doped with Ag impurities and functionalized with a layer of PV4D4 polymer.

2. Materials and Methods

2.1. Production Method

The Solution Chemical Synthesis (SCS) method was used to obtain ZnO sensors doped with Ag impurities. For this purpose, high-purity (>99.999%) ZnSO₄ and NaOH solutions were used. Initially, the ZnSO₄ salt was dissolved, then the NaOH was dissolved. The dissolved NaOH solution was gradually added to the dissolved ZnSO₄ solution, and after the final solution was obtained, the Ag impurities were also added. After this, the process of depositing Ag-doped ZnO began on a glass substrate measuring 75 × 25 × 1 mm. Throughout the deposition process, the solution was stirred using a magnetic stirrer. After the ZnO was deposited, it was subjected to a hot air flow for 30 s. A more detailed description of this method is provided in previous works [36,37].

Subsequently, the sample with an approximately 25 nm thin polymer layer of PV4D4 was covered on top, as previously reported in [32,38–40], using the monomer 1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetrasiloxane (V4D4, abcr, Hadfield, UK, 97%) and the initiator perfluorobutanesulfonyl fluoride (PFBSF, Fluorochem, Karlsruhe, Germany, 95%).

The sensing properties of the sensor upon exposed to various gases (hydrogen, 2-propanol, ethanol, acetone, ammonia) at a concentration of 100 ppm were evaluated using a Keithley 2400 sourcemeter (from Tektronix, Cleveland, OH, USA) connected to a computer with LabView interface software version 17.0f2 (from National Instruments, Austin, TX,

USA). The sensor respons S was calculated from the obtained results using the following formula [41,42] where G is the conductivity in different environment (gas or air):

$$S = \frac{\Delta G}{G_{air}} \times 100\% = \frac{G_{gas} - G_{air}}{G_{air}} \times 100\% \quad (1)$$

As additional comparison parameters for the sensor's performance, the response and recovery time were selected and defined as the time required for the response to increase from 10% to 90%, and decrease from 90% to 10%, respectively.

2.2. Morphology

The morphology of deposited films was investigated using scanning electron microscope (SEM) Zeiss Supra 55VP (from Carl Zeiss AG, Oberkochen, Germany). Structural properties have been investigated using Rigaku X-ray diffraction (XRD) (from Rigaku Corporation, Akishima, Japan).

SEM was used to study morphological properties of films, with the results presented in Figure 1. In Figure 1a are presented low-magnification SEM images of ZnO:Ag observing interconnected nanostructures, namely columnar grains of various sizes. At higher magnification (Figure 1b) there were observed nanodots, which are distributed on the surface of the columnar grains. The size of the nanodots was ~20–40 nm, determined using microscope software tools. The nanodots exhibit independence from one another, demonstrating either interconnection or percolation pathways.

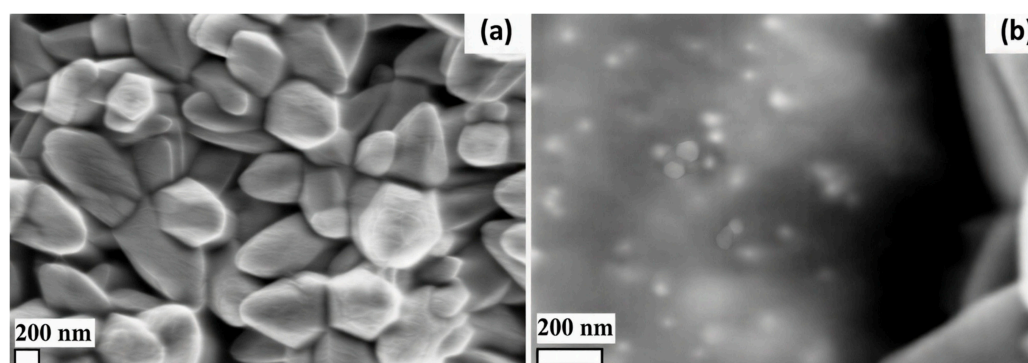


Figure 1. SEM images of ZnO: Ag films at (a) low and (b) high magnification.

Polymer coating was carried out via iCVD, as previously reported in various studies [31,32,39,40,43,44]. V4D4 monomer and a PFBSF (initiator), were introduced into a custom-built reactor in vapor phase. At the heated filament array, pyrolysis, the initiator generates free radicals. These free radicals react with the vinyl groups of the monomer, initiating chain growth polymerization and the incorporation of additional V4D4 molecules into the growing polymer network. Since V4D4 contains four vinyl groups, a highly crosslinked film is obtained after polymerization.

Thus, the fabricated thin-film polymer contributes to enhancing selectivity and sensitivity towards various analytes, which in the case of this paper was hydrogen. Also, previous works demonstrated that it offers protection from a humid atmosphere as well [31,32,45]. This type of polymer growth was selected as it enables a conformal coverage of large-area surfaces and complex geometries.

3. Results

Figure 2 shows the comparison of response limits to 100 ppm of different analytes. As it can be observed, hydrogen is the most predominant in the means of selectivity through

all tested operating temperatures. The highest registered response for hydrogen gas is 23% at the operating temperature of 350 °C.

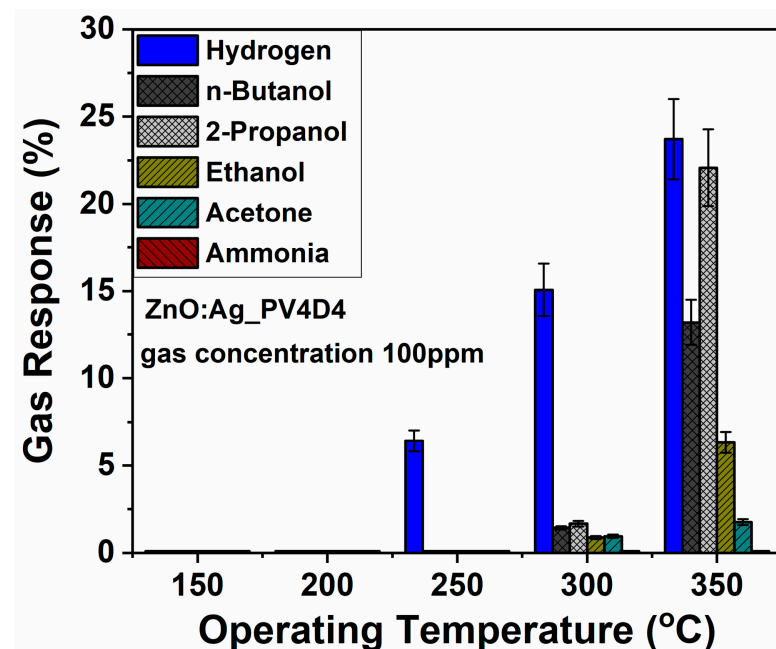


Figure 2. Sensor response of ZnO:Ag, coated with thin film of PV4D4 polymer on top, to several analytes (hydrogen, 2-propanol, ethanol, acetone, ammonia).

Figure 3a shows the dynamic characteristic of the ZnO:Ag-based sensor coated with a PV4D4 polymer on top with a response to 100 ppm hydrogen gas at operating temperatures of (a) 250 °C and (b) 300 °C, respectively. Thus, it can be observed that the higher the operating temperature, the better the result for H₂ gas detection is shown. At 300 °C, the response was $S \approx 14\%$; and at 250 °C, $S \approx 6\%$. Another very important property of the sensor structure is its response and recovery times. Comparing Figure 4a with Figure 4b it is observed that at OPT-300 °C the response and recovery times are shorter.

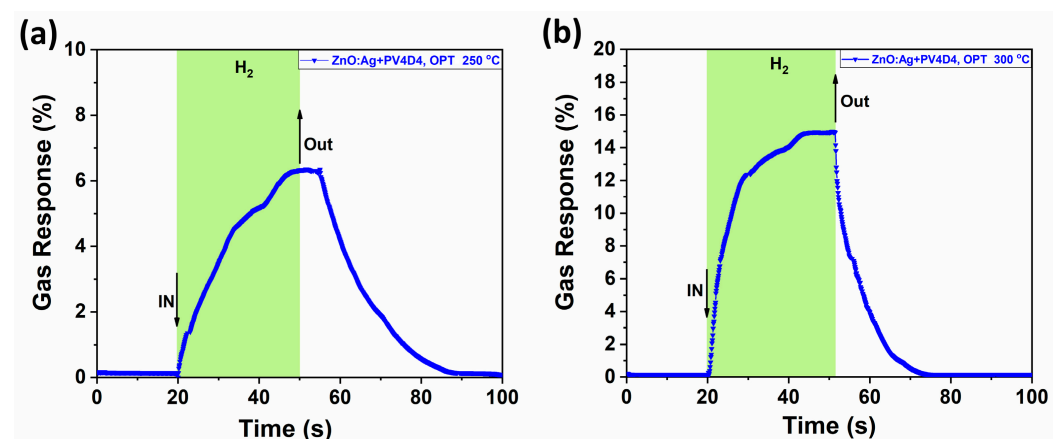


Figure 3. (a) Dynamic response of ZnO:Ag coated with PV4D4 to H₂ gas at OPT of 250 °C. (b) Dynamic response of ZnO:Ag coated with PV4D4 to H₂ at OPT of 300 °C.

Figure 4a shows the dynamic characteristics of ZnO:Ag structures coated with a layer of PV4D4 polymer for different gases (hydrogen, n-butanol, 2-propanol, ethanol, acetone). The figure shows that the shortest response and recovery times are for hydrogen molecules. The figure also shows that the recovery times for the other gases are longer than for hydrogen. Figure 4b shows the volt-ampere characteristics of ZnO:Ag structures coated

with a PV4D4 polymer layer. The figure shows that the characteristics obtained are typical of ohmic contact, and the highest current values are observed at an operating temperature of 350 °C.

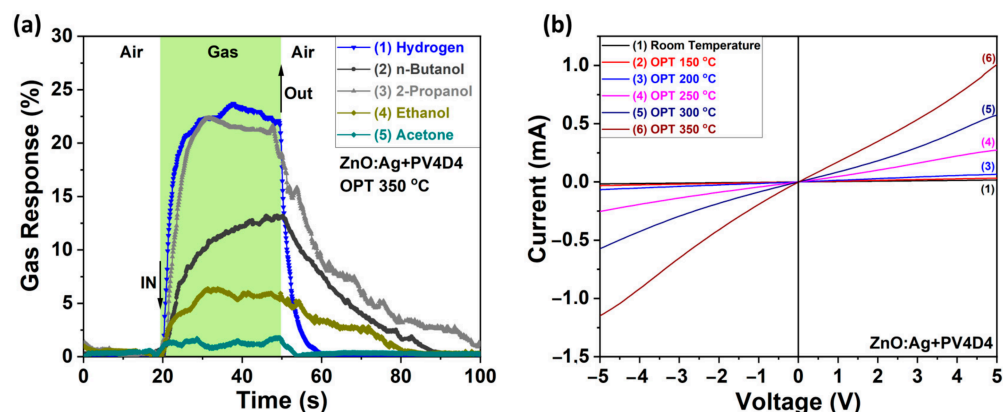


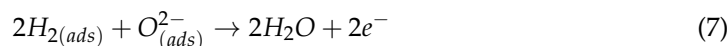
Figure 4. (a) Dynamic characteristics of ZnO:Ag structures coated with a layer of PV4D4 for different gases. (b) Volt-ampere characteristics of ZnO:Ag structures coated with a PV4D4 polymer layer.

4. Discussion

For a gas sensor, the gas detection mechanism plays an important role in describing its behavior. In the specialized literature, it is described that adsorbed oxygen species on the sensor surface play a particularly important role in the gas detection mechanism based on ZnO sensors. The following oxygen species are specific for different operating temperatures (O^- for the temperature range 100–300 °C and O^{2-} for operating temperatures above 300 °C) [45,46]. This process can be described using the following chemical equations:



When oxygen molecules in the form of O^{2-} interact with hydrogen molecules, H_2O molecules are formed, and electrons are released. This process can be described using the following chemical equations [32]:



Thus, by the combination of the proposed mechanism and polymer coating, it can be proposed that such a sensor, in its quality, can be used for further research, as well as for various industrial and medical applications where concentrations of 100 ppm of H_2 are used.

5. Conclusions

Following the completion of this work, it can be concluded that the use of cost-effective methods for obtaining gas sensors, followed by the deposition of a top-layer PV4D4 polymer film, successfully yielded a hydrogen-selective sensor structure operating efficiently within a temperature range of 250–350 °C. The sensor structure demonstrated a peak response amplitude of $S \approx 14\%$ and significantly shorter response and recovery times

for hydrogen gas compared to other operating temperatures and tested volatile organic compounds. This parameter, rapid kinetic behavior, is one of the most important performance metrics of a gas sensor. These findings underscore the strong potential of this sensor architecture for practical deployment in rapid and efficient hydrogen detection systems.

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