NEW VAPOR DEPOSITED DIELECTRIC POLYMER THIN FILMS FOR ELECTRONIC APPLICATIONS

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Abstract. Dielectric materials are of great interest in a vast amount of applications ranging from cable insulation to advanced electronic devices. The emerging trend of device miniaturization is creating an increased demand for dielectric thin films that can be produced precisely on the nanometer scale. In addition, special mechanical properties are often required, for example in the field of flexible organic electronics. Polymers are first-choice materials for this purpose. However, it is extremely difficult to produce precise nanoscale thin films, which have a low defect density and are free of e.g. residual solvent, by wet chemistry approaches. Initiated chemical vapor deposition (iCVD) is a solvent-free polymer thin film deposition process which can be used to produce high quality dielectric thin films with nanoscale control and circumvents thus these problems. This work demonstrates the versatility of the iCVD process in the field of electrical applications by some new application examples of iCVD. By adding e.g. a hydrophobic organosiloxane thin film on columnar zinc oxide (ZnO:Fe) gas sensing structures there was a change in the selectivity from ethanol to hydrogen, as well as improved performance at high humidity level. The modified sensors can thus be used in humid ambient, especially for breathing tests, which can lead to the diagnosis of some diseases by cutting edge non-invasive approaches.

Rezumat. Materialele dielectrice prezintă interes pentru numeroase aplicații, de la izolarea cablurilor până la dispozitive electronice avansate. Tendința emergentă de miniaturizare a dispozitivelor creează o cerere crescută pentru pelicule subțiri dielectrice care pot fi produse exact la scara nanometrică. În plus, sunt adesea necesare proprietăți mecanice speciale, de exemplu în domeniul electronicii organice flexibile. Polimerii sunt materiale de primă alegere în acest scop. Cu toate acestea, este extrem de dificil să se producă filme subțiri precise la scară nanometrică, care au o densitate scăzută a defectelor şi sunt lipsite de solvent rezidual, prin abordări ale chimiei umede. Depunerea chimică de vapori inițiată (iCVD) este un proces de formare a peliculei subțiri de polimer fără solvenți care poate fi utilizat pentru a produce pelicule dielectrice de înaltă calitate cu control la scară nanometrică şi eludează astfel aceste probleme. Această lucrare demonstrează versatilitatea procesului iCVD în domeniul aplicațiilor electrice prin câteva exemple noi de aplicare a iCVD. Prin adăugarea, spre exemplu, a peliculei subțiri de organosiloxan hidrofob pe structurile de detectare a gazului de oxid de zinc columnar (ZnO:Fe), se atestă o schimbare a selectivității de la etanol la hidrogen, precum şi o performanță îmbunătățită la un nivel ridicat de umiditate. Senzorii modificați pot fi astfel utilizați în mediu umed, în special pentru teste de respirație, care pot duce la diagnosticarea unor boli prin abordări non-invazive de ultimă oră.

Cuvinte cheie: *polimeri, depunere chimică inițiată de vapori, materiale electronice, dielectrice, electreți, senzori de gaz.*

1. Introduction

The current advent of polymer thin films enabled by the combination of chemical vapor deposition (CVD) with organic chemistry provides a variety of new pathways in the field of organic electronics and electronic applications. In particular, oxidative chemical vapor deposition (oCVD) and initiated chemical vapor deposition (iCVD) developed by Gleason et al. facilitate the precise deposition of new electronic materials from a few nanometers to several micrometers in film thickness [1]. While in oCVD a step growth reaction allows the preparation of conjugated polymers such as PEDOT or PANI [2], iCVD usually results in insulating films due to the underlying free radical polymerization. In the latter, the polymer backbone is sp3-hybridized and the polymer films can be used as dielectrics. Due to the solvent-free nature of iCVD, the dielectric thin films are of very high quality and have no defects that accelerate e.g dielectric breakdown [3]. Thanks to the CVD-typical growth character, the iCVD process can easily be scaled up and integrated into modern microelectronic process lines. A schematic illustration of the iCVD process is shown in Figure 1(a). The respective monomer (M) and initiator (I) are introduced to the reactor. The reactor is equipped with a heatable filament array, located above a sample stage, which is cooled to room temperature. The monomer molecules adsorb at the substrate stage and once the filaments are heated the initiator molecules are decomposed into free radicals (R). These radicals meet e.g. a vinyl groups of the adsorbed monomer molecules. They start a free radical polymerization, which follows similar kinetics like the well-known kinetics for wet chemistry free radical polymerization [4] in order to produce high quality polymer thin films at the substrate surface.

Figure 1(a) shows schematic design of the iCVD process. It is shown that monomer (M) vapor and initiator (I) vapor are introduced to a hot wire CVD (HWCVD) reactor at the same time. Afterwards, thermally decomposed I results in free radicals (R), which initiate a free radical polymerization with adsorbed M. Figure 1(b) shows possible monomers and initiators

for dielectric thin films. In this work, some application examples of different tailored dielectric iCVD films are shown to demonstrate the versatility of the process in the field of electrical applications.
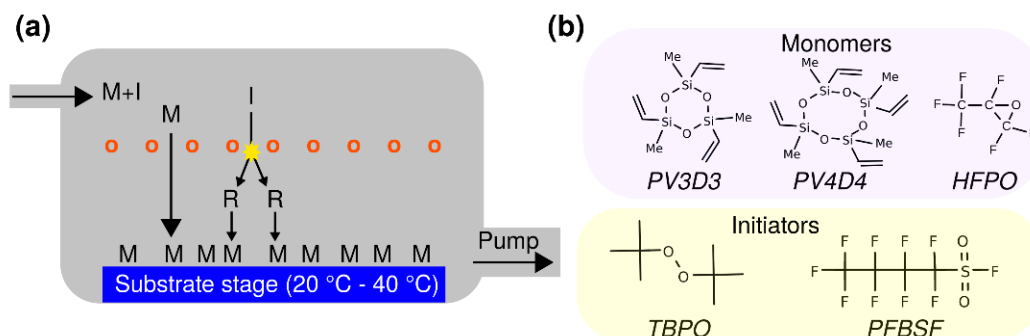

Figure 1. (a) Schematical illustration of the iCVD process. Monomer (M) vapor and Initiator (I) vapor are introduced to a hot wire CVD (HWCVD) reactor. Thermally decomposed I results in free radicals (R), which initiate a free radical polymerization with adsorbed M. (b) Possible monomers and initiators for dielectric thin films.

2. Experimental details

All polymer thin films reported in this study are deposited via iCVD. A home-made ring-inlet iCVD reactor explained in our previous works has been used for this purpose [3, 7, 8]. The hydophobic PV3D3 thin films have been obtained by the combination of the monomer V3D3 with the initiator PFBSF. The electrostatic potential maps have been illustrated using Jmol. The V3D3 and V4D4 molecules have been geometry-optimized employing density functional theory (DFT) at the B3LYP/ccPVDZ level for this purpose. The reported gas sensing structures have been obtained initially by the chemical deposition of solutions (SCS) method, which yields a nanostructured ZnO:Fe film. The film was deposited on a glass substrate (75 mm × 25 mm × 1 mm). The obtained ZnO, ZnO:Fe nanostructured films have been thermal annealed (TA) in furnace for 2 hours at 650 $^{\circ}$C in normal ambient. A more detailed description is given in our previous works [5, 6]. An ultra-thin deposition of CuO/Cu_2O with a thickness of 18 nm was grown on the ZnO:Fe film with a custom setup with DC and RF magnetron. After this step a heat treatment was applied to the samples in a furnace at 420 °C for 30 minutes. In the next fabrication step 25 nm of the hydrophobic PV3D3 thin film was deposited on top of the sensors. Finally, Au contacts were sputtered on top of the samples through a meandering mask [9]. For the sensor performance measurements, a computer-controlled Keithley 2400 sourcemeter at 0.2 V applied bias voltage was used. The details on the sensing experiments can be found in our previous works on gas sensing studies [10, 11].

3. Results and Discussion

The application of iCVD thin films as dielectric layer is reported by many authors, especially organocyclosiloxanes, like PV3D3, PV4D4 and fluoropolymers, have demonstrated excellent performance [12-14]. The chemical structure of possible monomers and initiators which yield excellent dielectric properties are presented in Figure 1(b). The polymer films can also be deposited on flexible substrates, as shown in the digital photograph in Figure 2(a), were we use a dielectric iCVD thin film as part of a flexible electronic device. Figure 2(b) shows a schematic representation of trap states, which are positioned between the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) bands of the polymer. Figure 2(c) illustrates calculated electrostatic potential maps of geometry-optimized V3D3 and V4D4.
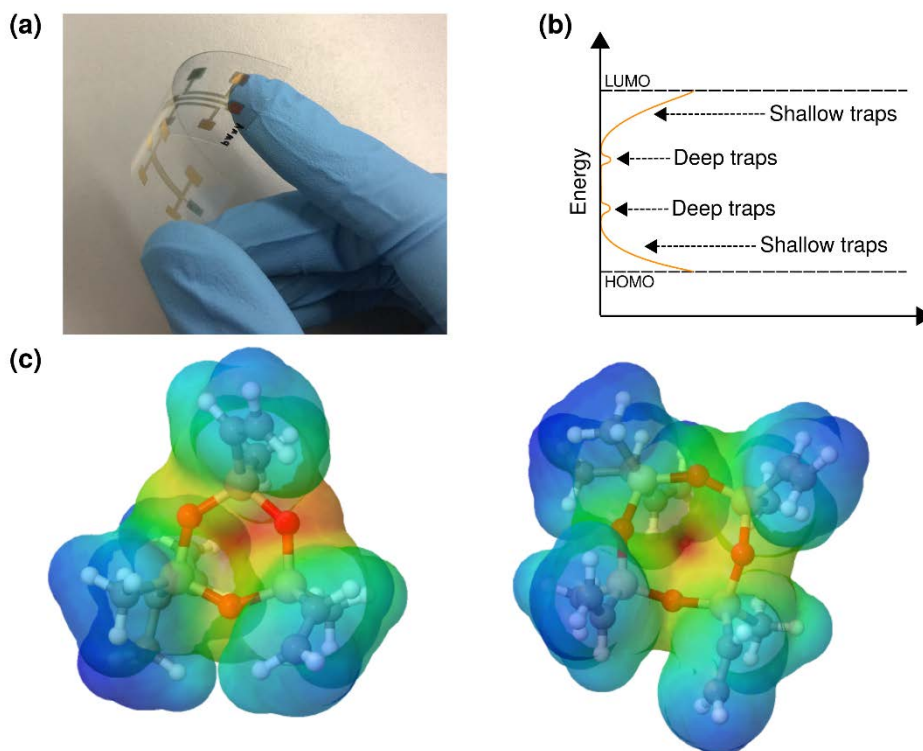
Figure 2. Photograph of the application of an iCVD thin film as part of a flexible electronic device, produced in our lab (a). Schematic illustration of trap states, which are located between the HOMO and LUMO bands of the polymer (b). Calculated electrostatic potential maps of geometry-optimized V3D3 and V4D4 (c).

The iCVD coatings are thus also highly attractive as dielectric thin films for flexible organic electronics. One further argument for using iCVD is the high film quality, because no organic solvents are used during the fabrication of the polymer thin films. Residual solvent and defects can cause e.g. conductive paths and this lowers the dielectric breakdown strength of the material. For dielectric iCVD films the breakdown strength has been found to be extremely high, because of the low defect density and high film quality without residual solvent molecules [3]. Since dielectric iCVD films exhibit such high breakdown strength and film quality, we demonstrated the use of iCVD grown PTFE thin films as electret materials in a previous work [3]. Electrets are dielectric materials with a quasi-permanent surface charge. They can be produced by exposing the dielectric materials e.g. to a corona discharge in order to transfer excess charge to the material. The charge carriers are stored in trap states, which are located in the gap between the HOMO and LUMO of the polymer, as illustrated in Figure 2(b). Typically deep level traps provide enough depth to store the charge carriers over a long period of time. The result is a surface potential which can be applied for many different application fields like portable electret condenser microphones, energy harvesting, air filters or magnetic field sensors [15, 16]. However, the charged film is in a metastable state and at some point the charge will decay. A good electret is thus characterized by the fact that its charge decay is negligible compared to the device lifetime in which it is used. Fluoropolymers like polytetrafluoroethylene (PTFE) typically exhibit the best charge storage properties [3]. The unique adjustability of the functional groups in the iCVD process allows the electrical properties to be specifically tailored. These can also be correlated with theoretical calculations. In Figure 2(c), the calculated electrostatic potential maps of the above-mentioned V3D3 and V4D4 monomers are shown as an example.

In addition to the production of such new tailored polymer thin films for electronic applications by iCVD, it is also possible to functionalize existing electronic devices. As an example, we recently demonstrated the functionalization of metal-oxide gas sensors with an ultra-thin hydrophobic cyclosiloxane-type of polymer [5]. In this previous work we have reported data for $CuO/Cu_2O/ZnO:Fe$ heterostructures, which were as-grown and Rapid Thermal Annealed (RTA) for 60 s at 650°C. After the deposition of the ultra-thin hydrophobic thin film via iCVD using the monomer V3D3 and the hydrophobic initiator PFBSF we were able to tune the selectivity of the gas sensors to hydrogen gas. Furthermore, we were able to operate the sensors at high humidity level, which has been a major problem for metal-oxide gas sensors before. Also, new metal-oxide $CuO/Cu_2O/ZnO:Fe$ heterostructures, which have been thermally annealed (TA) at 650°C for 2h in a furnace in air, can be applied as gas sensors. Scanning electron microscopy (SEM) images of these new structures are shown in Figure 3.
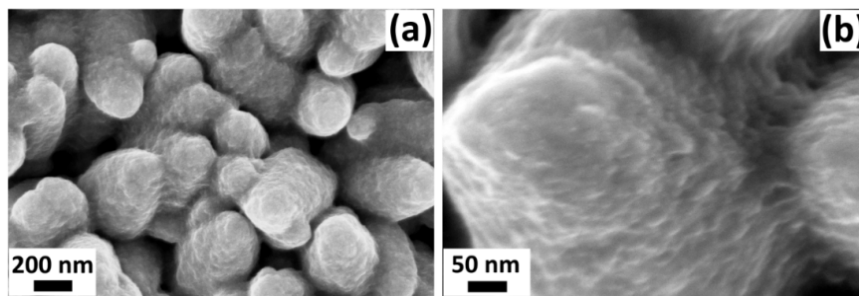

Figure 3. SEM images of $CuO/Cu_2O/ZnO:Fe$ heterostructures after thermal annealing at 650 °C for 2 h at low (a) and high magnifications (b).

From Figure 3 can be concluded that the samples are composed of highly packed columnar-type grains of ZnO:Fe with the CuO/Cu_2O layer on the surface. The structures cover the glass substrate completely. The increasing surface/volume ratio is beneficial for sensor applications.

The gas sensors shown in Figure 3 are coated with 25 nm hydrophobic PV3D3, similar like in our previously reported study [5]. PV3D3 layer was deposited on top of such sensor structure to protect it from the effect of environment and especially from the high relative humidity, which affect the sensor performances seriously. Figure 4 shows the XRD patterns of the samples.

The formation of hetero-junctions is evidenced by the coexistence of the phases of CuO, Cu_2O and ZnO, so that at the values 2θ of 32.45°, 35.3°, 46.15°, 65.4° and 68.05°. The reflections *hkl* were found for the copper oxide CuO (Tenorite) with the Miller planes of (-110), (-111)/(002), (-112), (022) and (113), respectively. These results clearly prove crystal quality and no changes due to iCVD process. Furthermore, the diffractions from the gold layer, Au (111) and Au (220), can be observed, which was deposited on top of the sensor structure as an electrical contact.

Reflections at 2θ of 34.35°, 34.35°, 47.95°, 56.75°, 62.2° and 66.45° with the Miller planes of (002), (102), (110), (103) and (200), respectively, are attributed to zinc oxide ZnO, according to the card (pdf # 36-1451) Zincite syn. In addition, reflections of iron oxide Fe_2O_3 are observed in Figure 4, which indicate the doping of zinc oxide with iron impurities. After thermal treatment Fe_2O_3 is found according to the card (pdf # 89-0599) Hematite syn with Miller indexes (104), (202), (024) and (125) at the 2θ angle of 33.45°, 43.45°, 49.05° and 67.1°, respectively.
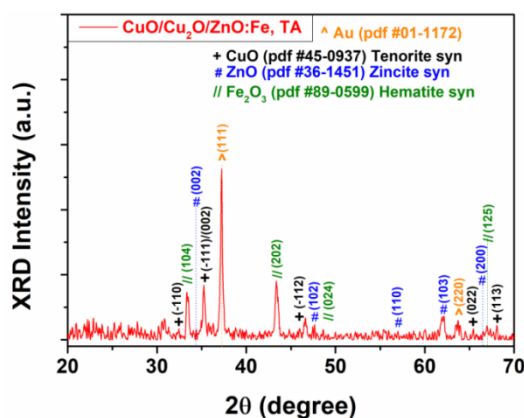
Figure 4. XRD pattern of CuO/Cu$_2$O/ZnO:Fe heterostructures thermally annealed at 650 $^\circ$C and contacted with Au on top as electrical contacts.
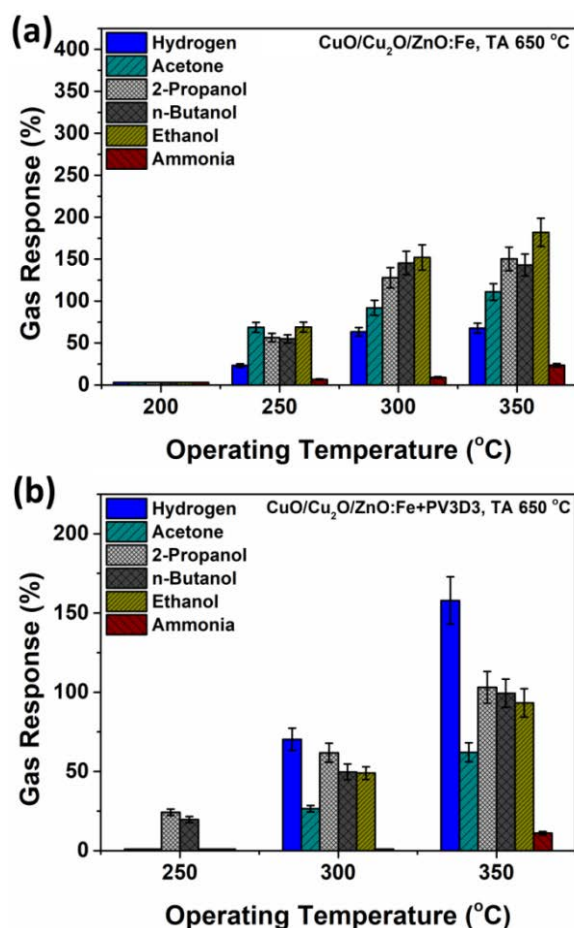

Figure 5. The dependence of the gas response (hydrogen, acetone, 2-propanol, n-butanol, ethanol and ammonia) versus working temperature for (a) uncoated heterostructured samples and for (b) hydrophobic PV3D3 coated samples.

As mentioned above, reflections at 2θ of 37,25 ° and 63,75 ° in Figure 4 are assigned to metallic Au, which originate from the sputtered contacts during device fabrication.

In order to test the final sensor performance with and without the polymer coating we performed gas response measurements with respect to various gas and volatile organic compounds in normal ambient. The results of the measurements of the gas response versus operating temperature are shown in Figure 5.

The sensors have been tested with hydrogen gas, acetone, 2-propanol, n-butanol, ethanol and ammonia vapor at different working temperatures. The results for thermally

annealed $CuO/Cu_2O/ZnO{:}Fe$ heterostructures without the PV3D3 are shown in Figure 5(a). Results from the sample set of thermally annealed $CuO/Cu_2O/ZnO{:}Fe$ hetero-structures with additional PV3D3 coating are shown in Figure 5(b) for comparison. Figure 5(a) reveals that the highest gas response can be observed for ethanol vapors. The growth of the hydrophobic PV3D3 on top of the sensor surface shows a shift of the selectivity to hydrogen gas, similar like our observations for the samples from our previous work [5]. The highest response can be obtained at an operating temperature (OPT) of 350 °C with a response value of ~165%. Figure 6(a) illustrates the dynamic response of the uncoated $CuO/Cu_2O/ZnO{:}Fe$ heterostructures to ethanol vapors in normal ambient. The dynamic response for hydrogen of the PV3D3 coated heterostructures is shown in Figure 6(b), which proves that selectivity was changed from ethanol vapor to hydrogen gas and pulses are reproducible during all experiments.
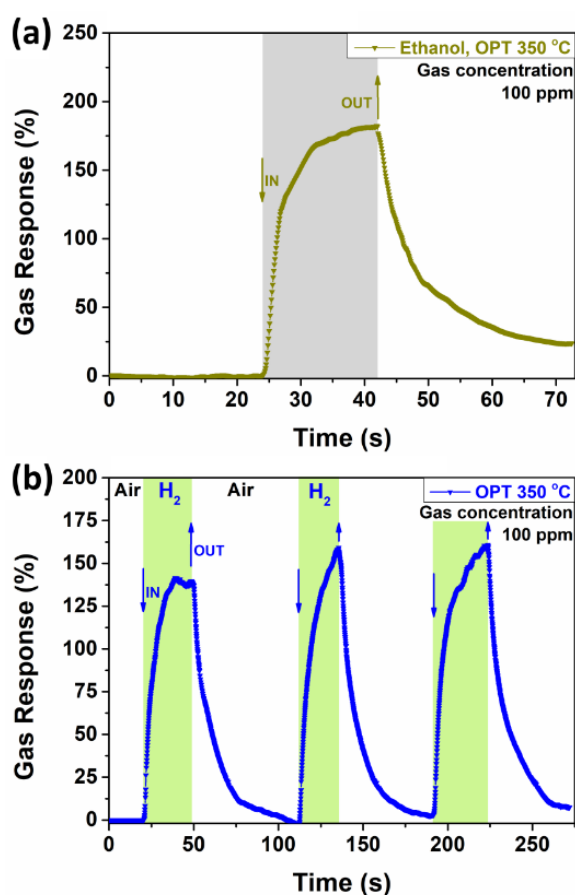

Figure 6. Dynamic response of the $CuO/Cu_2O/ZnO{:}Fe$ heterostructures thermally annealed at 650 °C for 2 h in air: (a) to ethanol vapors (sample set without PV3D3) and (b) to hydrogen gas (sample set with PV3D3 layer on top of the sensor) at optimal operating temperature of 350 °C measured in normal ambient.

Both measurements have been performed at an OPT of 350 °C and data recorded in normal ambient. The results in Figure 6 demonstrate that uncoated as well as coated heterostructures show quite high response values, when they are exposed to the respective gas. Furthermore, a very good repeatability can be observed in Figure 6(b). The sensors show a complete restoration of the response after each gas pulse.

The current-voltage (I-V) curves of the uncoated as well as coated heterostructures are shown in Figures 7(a) and 7(b), respectively. Figure 7a shows the electrical measurements for an uncoated heterostructure $CuO/Cu_2O/ZnO{:}Fe$ and Figure 7b shows the measurements for a

PV3D3-coated heterostructure on glass substrates. The measurements have each been performed at different OPT as indicated in the graphs and numbered accordingly. The electrical measurements reveal that the I-V curves for all heterostructures show Ohmic behavior at each of the targeted operating temperatures. This demonstrates that the sensors work correctly even with the additional 25 nm polymer coating on top of the heterostructure. As observed in the measurements above, the selectivity of the covered sensors has shifted towards hydrogen gas due to the additional PV3D3 coating, similar like in our previous study.
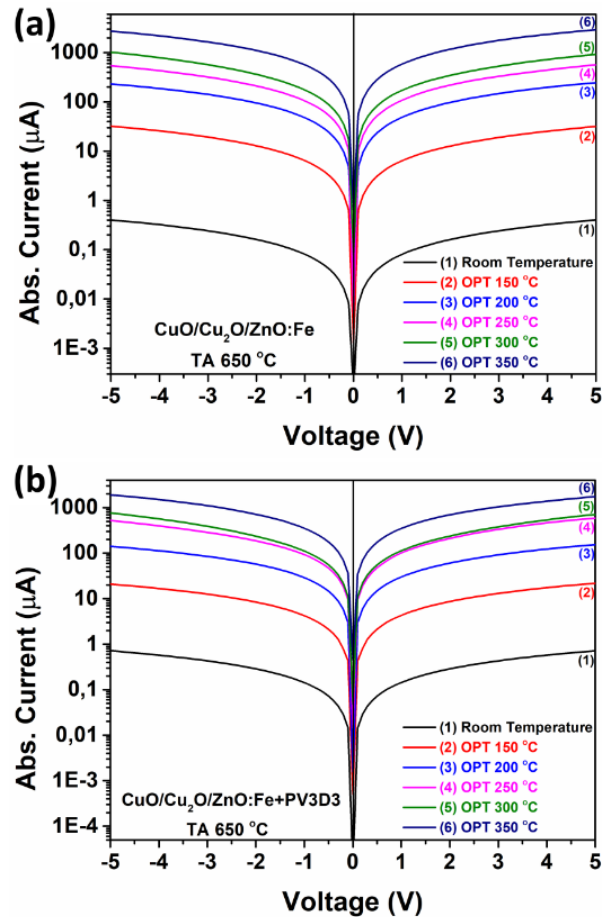

Figure 7. Current-voltage characteristics of the $CuO/Cu_2O/ZnO$:Fe heterostructures thermally annealed at 650 °C: (a) without PV3D3; and (b) covered with PV3D3 layers.

Dynamic response measurements at different humidity levels have been performed in order to investigate the influence of moisture or value of relative humidity RH on the sensor performance and to identify if it is suitable for breath test, as an example. The obtained results are shown in Figure 8. Figure 8(a) illustrates the dynamic response to ethanol vapor for the uncoated heterostructures. The dynamic response for the PV3D3-coated heterostructures are presented in Figure 8(b) for hydrogen. The gas concentration was in both measurements 100 ppm and the measurements have been performed at 20 % and 65 % relative humidity each. Figure 8(a) shows that the response of the uncoated heterostructure to ethanol vapor decreases drastically by about 88 % at relative humidity of 65 % compared to the response at relative humidity of only 20 %. As expected, this indicates that the gas sensors no longer work reasonably in a high humid environment and can't be used for example in non-invasive breath tests. The dynamic response measurement for the PV3D3-coated heterostructure, which is shown in Figure 8(b), reveals that the response to hydrogen

decreases to about 1/3 of the original response in the high humidity environment. In addition, it is evident that pulses are still repeatable at higher humidity. This clearly proves the effect of the coverage with a PV3D3 thin film to protect the gas sensing samples from moisture. The performed experiments thus show, that the additional hydrophobic PV3D3 coating can also increase the stability of the heterostructure with regard to a high relative humidity, similar like in our previous study. Furthermore, it can also protect the sensing structures from the environment conditions.
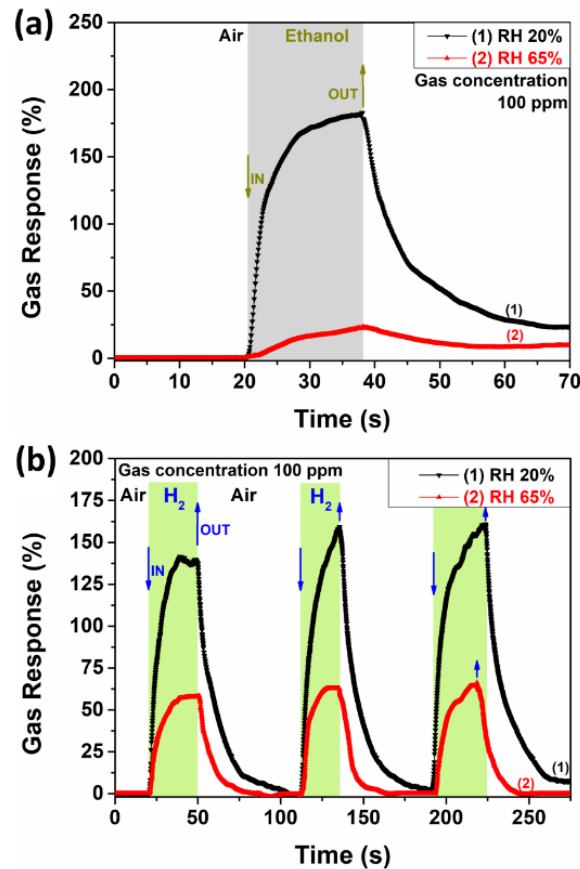

Figure 8. Dynamic response at operating temperature of 350 °C at different concentrations of relative humidity: (a) to ethanol vapors (samples without PV3D3 layer) and (b) to hydrogen gas (samples covered with PV3D3 layer on top).

4. Conclusions

Dielectric thin films deposited by iCVD can be used in a vast amount of electrical and electronic applications. We presented potential applications in organic electronics or as thin film electrets as examples. The individual tunability of the functional groups makes it possible to specifically adjust the electrical properties of the polymer thin films. Furthermore, in addition to creating new polymer thin film materials, existing devices can also be improved. The films can be used e.g. to protect electronic circuits or electronic devices. Following our previous study, we show as a real example, the use of hydrophobic coatings to protect semiconducting oxide-based gas sensors from moisture or high relative humidity. According to the presented results, $CuO/Cu_2O/ZnO:Fe$ heterostructures after thermal treatment at 650 °C for 2 h were successfully obtained in this current work. The structural results were confirmed by XRD data, with a granular morphology of columnar type confirmed by SEM. The heterostructures of $CuO/Cu_2O/ZnO:Fe$ with a thermal annealing regime in the

electrical furnace at 650 $^\circ$C for 2 h in ambient air are selective for ethanol vapors. After the deposition of a 25 nm hydrophobic PV3D3 thin film on the heterostructure, the selectivity changes from ethanol vapor to hydrogen gas. At the same time, the deposition of the PV3D3 thin film increases the stability at high relative humidity values, namely 65% RH in our experiments. A possible application field for the modified gas sensors in the future are biomedical applications. The sensors can here be applied for e.g. breath tests in non-invasive diagnosis, due to the fact that humidity does not significantly influence the response value to hydrogen gas in a high humid environment.

Conflicts of Interest. The authors declare no conflict of interest.

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