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Capacitive humidity sensors based on the dielectrophoretically manipulated ZnO nanorods

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ABSTRACT

In this paper, the capacitive relative humidity sensors based on dielectrophoretically manipulated ZnO nanostructure were constructed and studied. The humidity-sensitive ZnO nanorods were first synthesized by the thermal decomposition technique and deposited among the micromachined electrodes pairs via dielectrophoretic manipulation. The manipulated samples were detected as the capacitive sensors in which the nanostructured ZnO acted as the sensing elements. The capacitive humidity sensors were constructed by detecting the variations of the ZnO dielectric constant changing with the humidity environment. The results showed that the sensor had high humidity sensitivity, good stability, fast response/recovery time and well reproducibility. The dielectrophoresis manipulation can also improve the sensitivity of the sensor efficiently.

1. Introduction

The humidity sensors play an important role in daily life of human beings. Generally, the humidity sensors consist of the humidity sensitive element to sense the water contents and the transducer to transform the sensing stimuli into the useful electrical signals. So far many polymers or metal oxides are widely used as the sensing elements. Nanostructured oxides such as ZnO and SnO_2 had been proven the potential sensing element candidates which can improve the sensing performance remarkably [1–8]. Capacitive humidity sensors are promising devices for humidity detection due to the low power consumption, which favors their usage in hand-held devices [9,10].

Electrophoretic deposition has been used to fabricate the nanostructured materials. Unlike electrophoretic deposition, the dielectrophoretic manipulation can be used to arrange the micro/nanomaterials between the electrodes and improve the performance for humidity detection [11]. Thus, a novel capacitive humidity sensor based on nanostructured ZnO can be constructed by detecting the variations of the ZnO dielectric constant due to the adsorption/desorption of water contents. The sensing properties, such as the sensitivity, the reproducibility, and the stability were studied.

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2. Capacitive sensor characteristics

2.1. Sensing principle

Upon adsorption, the physical properties of nanostructured ZnO change owing to the adsorption of water vapor molecules. This leads to the change of dielectric constant. Assuming the validity of Henry's law for low analyte concentrations, the change in dielectric constant upon water vapor adsorption, $\Delta\varepsilon$, should be positive and expressed as:

$$\Delta\varepsilon \propto \varphi_{\mathrm{w}}(\varepsilon_{\mathrm{w}} - \varepsilon_{\mathrm{air}}) \quad (1)$$

where ε_{w} and $\varepsilon_{\mathrm{air}}$ are the dielectric constant of water and air, respectively, and φ_{w} is the amount of adsorbed water expressed as volume fraction. The variation $\Delta\varepsilon$ will lead to the change of sensor capacitance. Since the relative dielectric constants of water ($\varepsilon_{\mathrm{w}} = \sim 70$) is bigger than that of air ($\varepsilon_{\mathrm{air}} = 1$), the water vapor adsorption on ZnO will lead to the big change in dielectric constant, $\Delta\varepsilon$, i.e., the big capacitance change, ΔC. The introduction of nanostructured ZnO as sensing element will further amplify this effect since the nanostructured ZnO has the huge surface-to-volume ratio. All these are beneficial for the sensitivity improvement.

2.2. Experiment setup

The schematic diagram of humidity detection system can be found in Ref. [10]. The system consisted of three parts: the standard humidity generation, the 555 capacitance–frequency conversion circuit, and the data recording system.

The saturated aqueous solutions of LiCl, $C_2H_3KO_2$, $Mg(NO_3)_2$, K_2CO_3, KI, NaCl, KCl and $CuSO_4$ in a closed glass vessel at an ambient temperature of 25 °C were used, which can generate the corresponding standard humidity levels of 11.30%, 22.51%, 43.16%, 52.89%, 68.86%, 75.30%, 84.34%, and 97.6% RH, respectively [12]. In detection, the sensors were put into these vessels and the responses were recorded and analyzed.

The 555 capacitance–frequency conversion circuit can change the capacitance variations of the sensors into the frequency shifts.

The LabView virtual instruments DAQ PCI6221 (PCI6221, NI, USA) were used to collect the output frequency of the 555 IC multivibrator circuit in real-time. The controlled humidity environments were generated using saturated aqueous solutions in the closed glass vessel.

The entire setup for relative humidity sensors was maintained at $\sim 25 \pm 1$ °C in the cleanroom environment.

2.3. Sensor construction

Four kinds of gold electrode configurations, the labyrinth electrode array, the interdigitated electrode array, the castellated electrode array and the circle electrode array, were fabricated on oxide covered silicon substrates by optical lithography and labeled as (a), (b), (c), and (d), respectively. Nanostructured ZnO was fabricated as shown in Ref. [11]. Fig. 1 shows the results of nanostructured ZnO aligned and located into the gaps of microfabricated electrodes. The amount of ZnO between the electrode gaps can be adjusted by changing the parameters of the dielectrophoretic manipulation, such as the voltage, the applied frequency, and the concentration of the suspension used.

After dielectrophoresis, the silicon chips were fixed on a TO-8 base and lined by wire and silver conductive paint and formed the humidity sensors.

3. Results and discussion

ZnO nanorods located between the electrodes can be regarded as the absorbent for water vapor. When the relative humidity of surrounding increases, the ZnO nanostructure will adsorb the water vapor molecules and lead to the dielectric constant of ZnO increasing. This can be embodied by the capacitance increment, i.e., the decrease of output frequency.

3.1. The influence of different electrode configurations

The sensor samples indexed as 1, 3, 4, and 6 expressed the sensors employed the electrodes (a), (b), (c), and (d) in Fig. 1. The ZnO was manipulated by negative dielectrophoresis. For the samples with different electrodes, the initial frequency values (f_0) were different, implying that the different electrodes configurations result in the different capacitance values. The detection can proceed either by low to high or high to low humidity directions. In the low to high process, the initial frequency was defined as the sensor frequency measured at humidity level of 11.4% RH. In the testing, the humidity levels increase to 97.6% RH step-by-step. For the high to low process, the initial frequency was defined the sensor frequency measured at humidity level of 97.6% RH. In the testing, the humidity levels decrease to 11.4% RH step-by-step. Fig. 2 shows the frequency shifts (corresponding to the corresponding initial frequencies) and the relative humidity levels. The arrows in the figures indicated the humidity changing direction in testing.

For all four samples, the sensors had linear responses from relative humidity of 20–80% RH, except for sample 1. The sensitivity of humidity sensors can be defined as the slopes of the frequency–relative humidity curves. The sensitivity values are always negative, indicating that the frequency values will decrease with the increasing humidity. Among these four samples, the labyrinth electrode array had the highest absolute sensitivity value of 205.3 Hz/RH%. However the linearity of this electrode was apparently deteriorated when the relative humidity was higher than 80%.

The measured values were also linearly fitted and the results were summarized in Table 1.

3.2. The influence of with and without dielectrophoretic manipulation

Fig. 3 shows the comparison for the sensors without dielectrophoresis (sample 2) and with negative dielectrophoresis (sample 3), where the interdigitated electrodes were employed. It can be found that the sensitivity, 52.3 Hz/RH%, for sample 3 (with DEP), was bigger than 28.9 Hz/RH%, for sample 2 (without DEP). The sensors constructed by DEP show the improved sensitivity. The reason can be attributed to two aspects: one is that more ZnO will be positioned in the pairs via the DEP. This can be proved by SEM pictures in our previous paper [10]. The other one is that after DEP process the ZnO nanostructure can be manipulated along the external electric field. The aligned ZnO seem to increase the dielectric constant and lead to the bigger initial capacitance value compared to the un-manipulated sensors.

3.3. The influence of the negative DEP and the positive DEP

In our study, both the negative DEP and the positive DEP can be observed. And the sensors based on these two effects were constructed.

The comparison between the sensor with positive dielectrophoresis (sample 5) and the sensor with negative dielectrophoresis (sample 6) was also studied. The electrodes employed were the circular shape. The initial frequency values, i.e., the initial capacitance values, of these two sensors were very close. The sensitivity values of samples 5 and 6 were 4.9 Hz/RH% and 4.1 Hz/RH%, respectively. No novel difference between the sensitivity values can be found. Both the positive and negative dielectrophoresis manipulation can make the ZnO aligned along the electric line orderly. And the only difference is the ZnO location in the electrode gaps. It implied that the location of the ZnO nanostructures in the electrode gaps has little influence on the sensor capacitance, i.e., the little influence on the sensor characteristics.

3.4. Hysteresis

In hysteresis testing, the sensor was tested from low to high humidity level, following by testing from high to low humidity level. Fig. 4 shows the hysteresis curves of four sensor samples.

It is found that the responses along the humidity decreasing process (red points) were always lower than those along the humidity increasing process (black points) and the hysteresis loops can be observed. The hysteresis was induced by the adsorbed water which cannot be desorbed quickly. The hyteresis behavior of sensors were different. The sensor employing the labyrinth electrode showed the lowest hysteresis. This is probably due to the smallest fringing electric fields induced for this sample.

3.5. Repeatability and response time

Fig. 5 shows the frequency shift of sample 1 as a function of time for different relative humidity. Sample 1 worked with a humidity cycle of low-to-high and high-to-low step. From figure, it can be seen that the differences between the ascending and