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Micro humidity sensors based on ZnO–In_2O_3 thin films with high performances

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ABSTRACT

Micro humidity sensors are fabricated by radio-frequency sputtering ZnO and In_2O_3 thin films on SiO_2/Si substrates with interdigitated Pt signal electrodes. By adjusting the sensing film form, the micro sensors with a tiny area (1.36 mm × 0.55 mm) exhibit controllable humidity sensing properties. The best sample is obtained by sputtering two times of ZnO and one time of In_2O_3 on sensor active area (0.63 mm × 0.55 mm). The corresponding impedance changes by more than four orders of magnitude over the whole testing humidity range (from 11% to 95% relative humidity), and the response and recovery times are about 15 s and 40 s, respectively. High stability and good consistency are also observed based on the as-fabricated sensors. The improved and excellent humidity sensing properties are explained by the hetero junctions between ZnO and In_2O_3 thin films. High sensor performance and large-scale fabricating potentiality demonstrate that the micro humidity sensors are very promising devices for humidity detection which could be produced at industrial level.

1. Introduction

In recent ten years, the booming research on micro sensors has opened new perspectives to develop various automatic control devices [1–4]. Hitherto, many high performance physical or chemical micro sensors have been reported. But most of them are focused on gas sensors or temperature sensors [5,6]. Humidity sensors, even with very simple electrodes, are hard to be fabricated with microstructures [7–14]. This is because many humidity sensors are based on semi-conducting metal oxides (SMO) with alkali-dopants (such as K, Li, or Na) [15]. These dopants may hydrolyze and unable to recovery in small film areas [15,16]. Thus the stability and repeatable of many micro humidity sensors are unsatisfactory in practical applications.

For SMO humidity sensors, their electrical impedances decrease for increasing humidity due to the enhancement of the chemically or physically adsorbed water on sensor surfaces [17–19]. In low humidity ranges, the tips and defects of the sensing materials present a high local charge density and a strong electrostatic field, which promote water dissociation. The dissociation provides protons as charge carriers of the hopping transport. At high humidity, one or several serial water layers are formed on sensor surface, and electrolytic conduction takes place along with protonic transport, and becomes dominating in the transport-process [20]. Accordingly, the sensor performance is strongly related to the forms of materials, namely, grain size, surface area, dimension, as well as the dopants in the base materials [21]. Many scientific and technological efforts are applied to design and tailor sensing materials with various techniques [22–24]. Doping alkali-dopants in SMO has been proved to be a simple and effective way for humidity sensing enhancement [20–25]. But as-mentioned above, the sensors may suffer from poor physical and chemical stability due to the high solubility of these dopants (especially for sensors with small size functional films). Therefore, SMO humidity sensors without alkali-dopants may be very useful in the future sciences and applications.

In this paper, we present micro humidity sensors achieved by radio-frequency sputtering ZnO and In_2O_3 thin films on sensor substrates with a tiny area. ZnO and In_2O_3 are traditional SMO humidity sensing materials, and the related sensors have been investigated for more than three decades [26,27]. As no alkali-dopants used in the sensing films, the sensors exhibit very high stability and good consistency. Besides, radio-frequency sputtering is outstanding for large-scale fabrication, excellent compositional control, and good surface morphology. These characteristics make the sensors good candidates for fabricating micro humidity sensors with high performances at industrial level.

2. Experimental

2.1. Preparation and characterization of materials

ZnO and In_2O_3 thin films were deposited on sensor substrates by a radio-frequency sputtering system (JZCK-IVB, Shenyang). Sintered ceramic ZnO and In_2O_3 targets (purchased from

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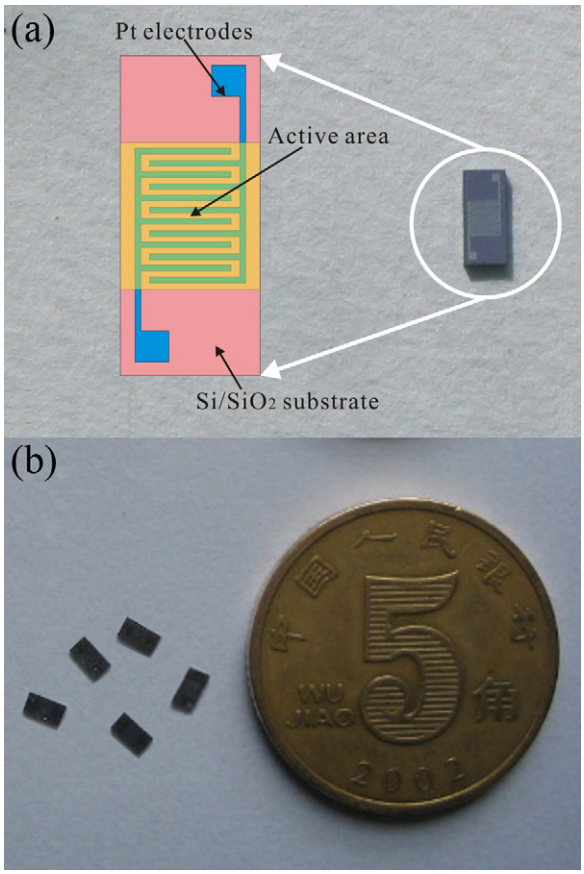

Fig. 1. Design and a photograph of the sensor substrates (a) and comparison of sensor substrates and a Chinese coin (b).

Beijing Huifangyuan Co. Ltd., Beijing) with 99.999% purity of 55 mm in diameter were employed as source materials. The target-to-substrate distances were kept at 6 cm. Before sputtering, the vacuum chamber was evacuated down to a base pressure of 1.0×10^{-3} Pa. High purity (99.999%) Ar and (99.999%) O_2 were introduced through separate mass flow controllers. The total pressure during sputtering was maintained at 1.0 Pa, and the O_2/Ar ratio was 1/10. The substrate temperature was controlled at 300 °C. The depositing frequency was 13.56 MHz and depositing powders for ZnO and In_2O_3 were 130 and 150 W, respectively. Before deposition, the target was pre-sputtered in O_2 + Ar atmosphere for 15 min to remove any impurity on the surface of the target.

Field emission scanning electron microscopy (FESEM) images were performed on a JEOL JEM-6700F microscope. X-ray diffraction (XRD) analysis was conducted on a Rigaku D/max-2500 X-ray diffractometer with Cu Kα radiation (λ = 1.5418 Å). Film thickness was measured by a step profiler (AMBIOS Technology INC XP-2).

2.2. Sensor fabrication

SiO_2/Si substrates were purchased from Dingjin Electronic Material Co. Ltd (Luoyang). Sensor substrates were obtained with following steps: (a) sputtering Pt metal film with the thickness of 1000 Å; (2) mask patterns transfer to the SiO_2/Si substrates by photolithography; (3) etching the Pt metal film to form signal electrodes. The sensor area was 1.36 mm × 0.55 mm, and the active area among interdigitated electrodes was 0.63 mm × 0.55 mm. The sensor substrate design and a photograph of the substrates are shown in Fig. 1(a). Fig. 1(b) shows the area comparison of several sensor substrates and a Chinese coin.

Table 1
Information of all samples.

Sensor name	Fabricating process (each sputtering time was 2 h)
Sensors (Z)	Sputtering ZnO only (1st)
Sensors (I)	Sputtering In_2O_3 only
Sensors (ZI)	Sputtering ZnO + In_2O_3 (2nd)
Sensors (ZIZ)	Sputtering ZnO + In_2O_3 + ZnO (3rd)
Sensors (ZIZI)	Sputtering ZnO + In_2O_3 + ZnO + In_2O_3 (4th)

Sensors were fabricated by sputtering ZnO and In_2O_3 thin films on sensor substrates with masks, and then calcining at 600 °C for 1 h. Five types of sensors, with different sensing film forms, are listed in Table 1, and the fabricating processes are also illustrated in Fig. 2 to explain the film structure.

2.3. Humidity sensing measurement

Humidity sensing properties were measured by a CHS-1T (Chemical humidity sensor-1 temperature) intelligent humidity sensing analysis system (purchased from Beijing Elite Tech Co., Ltd., Beijing). The voltage applied in our studies was alternating current (AC) 1 V and the frequency was 100 Hz. Two stretch probes were pressed on the sensor electrode-pins as the lead-wires. Response and recovery characteristics were tested by switching the sensor between two chambers with LiCl (corresponding RH value was 11% RH) and KNO_3 (corresponding RH value was 95% RH) saturated salt solutions, respectively [28]. Humidity ambiences for other tests were generated by using a double-flow humidity source in the analysis system. Two flows with different relative humidity (RH) (3% and 99% RH) were flowed into the test chamber with high precise control to form the target RH condition.

3. Results and discussion

Fig. 3(a) and (b) shows the SEM images of ZnO and In_2O_3 thin films, respectively. The morphologies of ZnO and In_2O_3 grains are found to be continuous and dense, and the crystallite diameters are found to be about 25 and 30 nm for ZnO and In_2O_3, respectively. For ZnO thin films, the XRD pattern (insert in Fig. 3(a)) exhibit the (0 0 2) and (0 0 4) peaks (consistent with the values in the standard card, JCPDS 36-1451) [23], indicating that the obtained films are polycrystalline with the hexagonal wurtzite structure. While all of the diffraction peaks in the inset of Fig. 3(b) can be indexed to the cubic In_2O_3 with lattice constant of a = 1.011 nm (JCPDS card no. 06-0416) [29]. No diffraction peaks from any other impurities are detected in these patterns, indicating the high purities of as-sputtered films.

For humidity sensors, impedance signals are usually applied to evaluate sensor performance in order to avoid the effects of polarization of adsorbed water on sensor surface [30–33]. Therefore, each type of sensors is tested in AC condition to describe the sensor response to changes in humidity. As shown in Fig. 4, under an electrical field at 100 Hz, sensors (Z) and (I) show obvious changes of impedance with respect to the humidity only in ranges higher than 54% RH. By depositing both ZnO and In_2O_3 films on sensor substrates, the sensor performance can be greatly enhanced. Sensors (ZI), (ZIZ), and (ZIZI) show much larger signal changes with better linear correlative curve, confirming that the complex film structures directly contributes to the increase of conductivity and the improvement of the linearity. The highest sensing performance is found for sensors (ZIZ), which present the best linearity and largest impedance change. In particular, the impedance of sensors (ZIZ) decreases linearly with increasing RH by four orders of magnitude with RH variation of 11–95%, exhibiting very high humidity sensitivity. Therefore, all the discussions below are focused on this type of sensors.

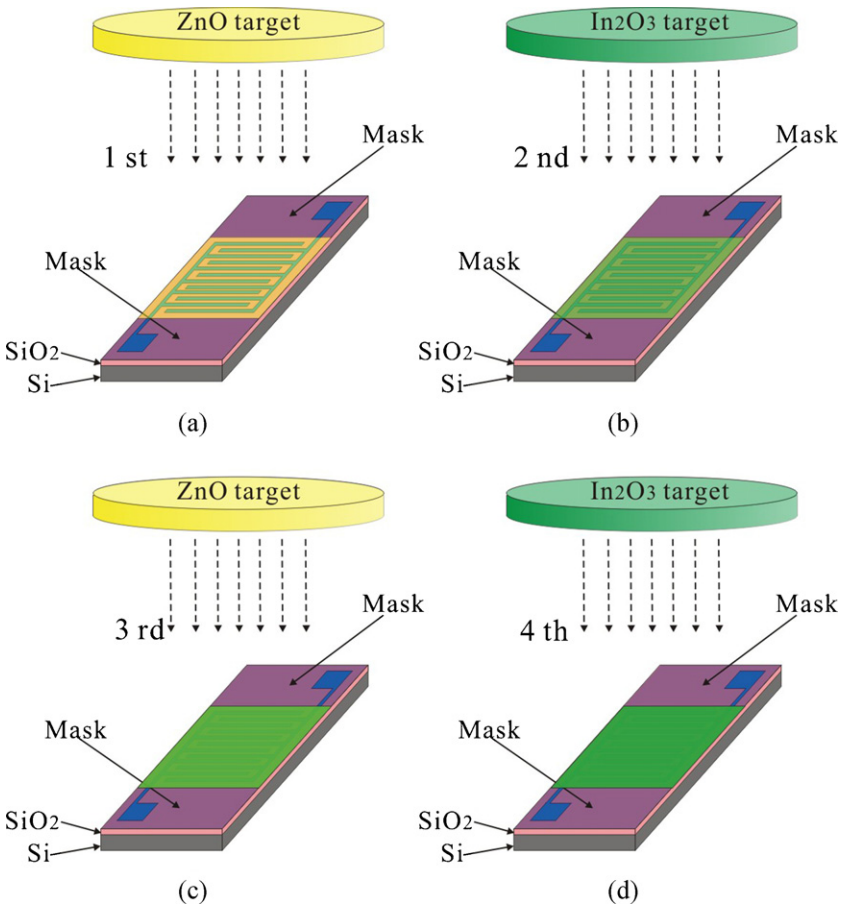

Fig. 2. Fabrication processes of micro humidity sensors with various film forms.

Response and recovery behavior is one of the significant features for estimating the performance of the humidity sensor. The time taken by a sensor to achieve 90% of the total current change is defined as the response time in the case of adsorption or the recovery time in the case of desorption. Quick response and recovery times are observed based for sensors (ZIZ), as shown in Fig. 5. The impedance of the sensors becomes stable within 15 s after exposure to 95% RH, and returns to the original values within 40 s after the tested humidity condition is replaced by 11% RH. Thus the response time (humidification from 11% to 95% RH) is about 15 s, and the recovery time (desiccation from 95% to 11% RH) is about 40 s. The recovery time is much longer than response time: in fact the desorption process of the absorbed water in the humid membrane is slower than the corresponding absorption process. Similar characteristics are also observed in many previous sensors [20,32].

Hysteresis is the time lag in the adsorption and desorption process, and is usually used to estimate the reliability of humidity sensors [20]. As shown in Fig. 6, the typical hysteresis of sensors (ZIZ) between absorption and desorption processes is less than 4% RH in the range of 11–95% RH, indicating a good reliability of sensors (ZIZ).

To test the long-term stability of humidity sensors (ZIZ), they were exposed to air for 60 days: afterwards their impedances were measured at various RH. As shown in Fig. 7, there is acceptable change in the impedances, proving the good stability of humidity sensors (ZIZ).

For each type of sensors, several samples were fabricated, and all results discussed from know on are based on the results of sensors exhibiting medium performances. Humidity sensors fabricated by artificial coating or printing, often do not present satisfactory consistency. Therefore, all the sensors performances were tested and Fig. 8 shows the impedance-RH relations from six sensors. The sensors almost show the same impedance values at every test conditions, which directly confirm the excellent consistency of the sensors (ZIZ).

The sensing mechanism of SMO related humidity sensors has been explained in many previous papers [34–40]. The change in impedance is primarily caused by the adsorption and desorption of the water molecules on the surface of the SMO films. Briefly, hopping transport of protons is the only possible conducting factor in low RH (only chemisorbed water molecules in this condition), thus the corresponding sensor impedance is very high (about 4000 kΩ in our cases) [35–37]. When the level of relative humidity grows, water vapor grows too. After the chemisorbed process is finished, subsequent layers of water molecules are physically adsorbed on the sensing film surface. Protons will travel freely in the continuous water layers, and synchronously, the physisorbed water will dissociate as $2H_2O \leftrightarrow H_3O^+ + OH^-$ due to the electric fields. Therefore, the electrolytic conduction occurs when H_3O^+ releases a proton to neighboring water molecules, which accept it while releasing another proton. This is known as Grotthuss chain reaction [38–40,35]. With further increasing RH, liquid water may be produced on the sensor surface and contribute to the conductivity. After depositing a type of SMO film on another type of film, the sensor performance has been effectively improved. This is mainly due to some hetero junctions formed between these films during the calcining process due to the outstanding activation of the In_2O_3 and ZnO nanoparticles prepared by radio-frequency sputtering [41,42]. Many more tips and defects will be produced among these hetero junctions, and the enhanced charge density and electrostatic

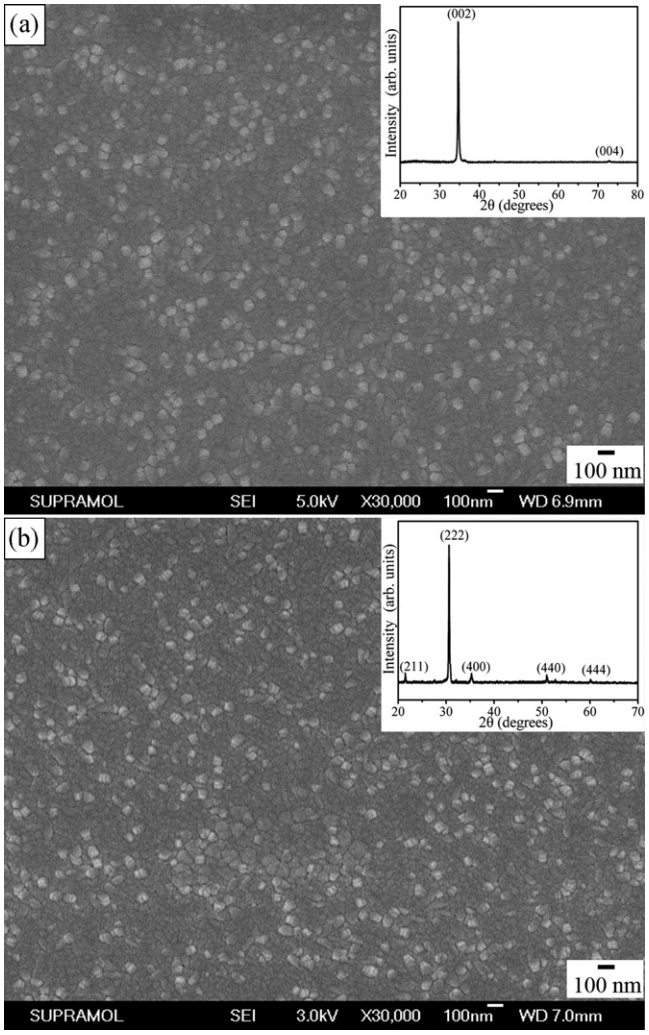

Fig. 3. SEM images of ZnO (a) and In_2O_3 (b) thin films (the inserts show the corresponding XRD patterns).

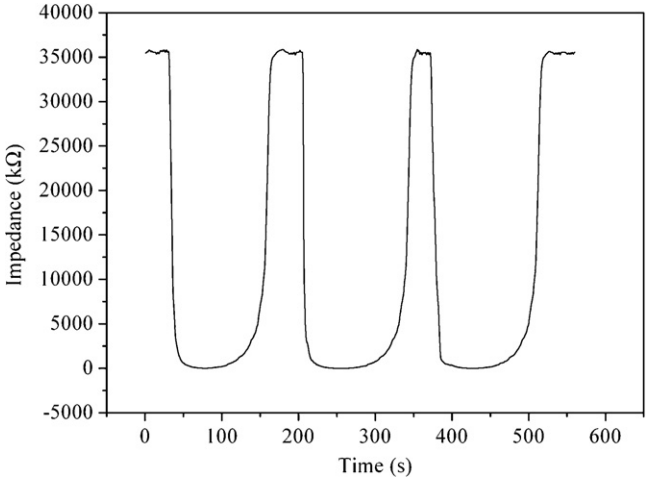

Fig. 5. Response and recovery characteristic of micro humidity sensors (ZIZ) from 11% RH to 95% RH.

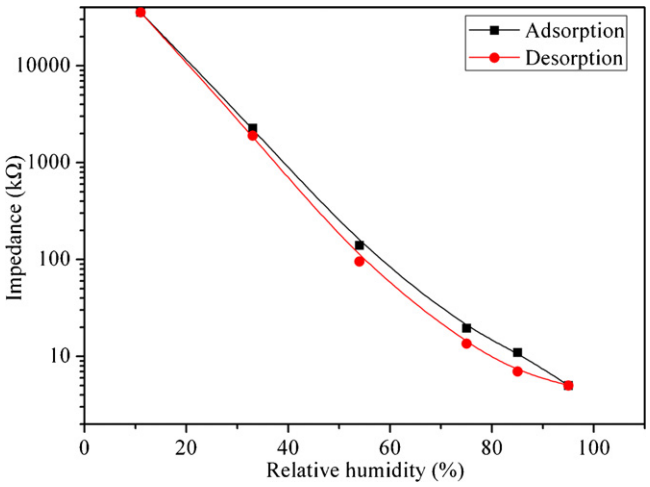

Fig. 6. Hysteresis of micro humidity sensors (ZIZ).

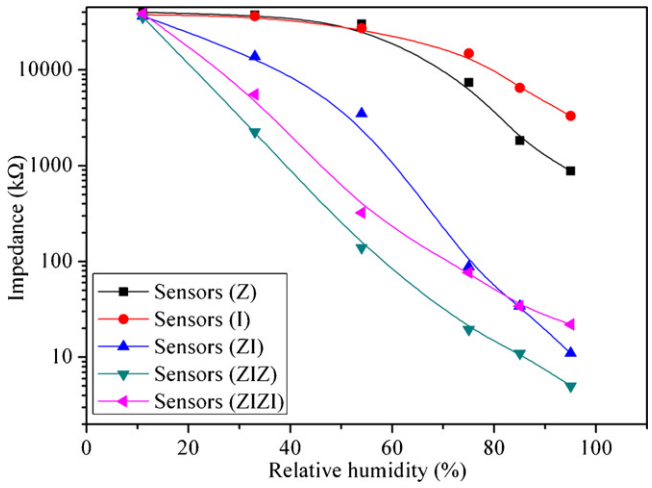

Fig. 4. Humidity sensing properties of micro humidity sensors with various film forms.

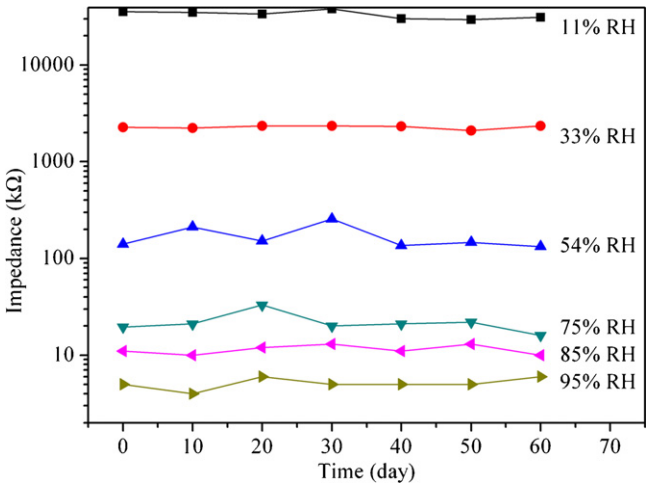

Fig. 7. Stability of micro humidity sensors (ZIZ).

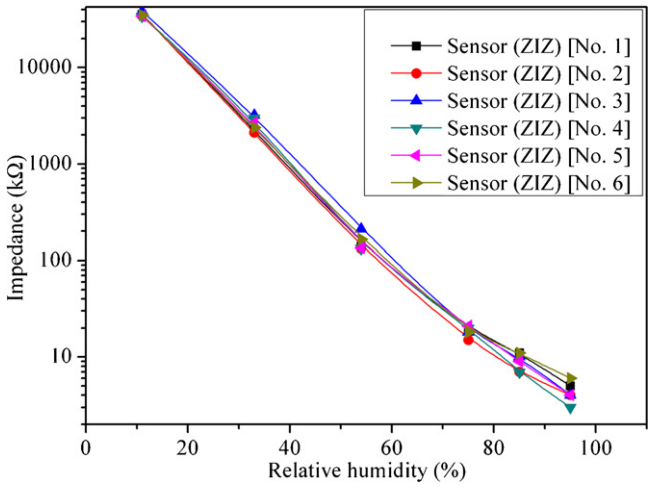

Fig. 8. Consistency of micro humidity sensors (ZIZ).

field are vital factors for high humidity performances [20]. On the other hand, when the sensor is up to humidity ambience, water molecules will permeate into the interface of these junctions, which lead to enormously changes in electrical signals [43,44]. Moreover, the decreased performance of sensors (ZIZI) can be explained by considering the thickness of the sensing film. Each layer of sensing film is measured to be about 300 nm, thus the sensors (ZIZI) with the film thickness of about 1.2 μm may unable to permeate water molecules to under layers, and this will lead eventually to decreased electronic signals.

4. Conclusion

In summary, micro humidity sensors with a tiny area (1.36 mm × 0.55 mm) are fabricated by radio-frequency sputtering ZnO and In_2O_3 thin films as the sensing materials. Among all the tested samples, sensors (ZIZ) exhibit the highest sensing properties. The impedance changes of this type of sensors is more than four orders of magnitude for 11–95% RH increase, and corresponding response and recovery times are about 15 s and 40 s, respectively. High stability and good consistency are also observed based on the fabricated sensors. The humidity sensing mechanism is provided by considering the film form. The tiny area and high performance make sensors (ZIZ) good candidates in sensor integration, and the sensing films without alkali-dopants may provide another approach for humidity sensor design and fabrication.

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