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Electrospun titanium dioxide nanofiber humidity sensors with high sensitivity

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Abstract

Titanium dioxide nanofibers were synthesized using electrospinning technique. The nanofibers were porous with an average diameter and length of ~150 nm and 200 μm, respectively. Humidity-sensing devices were fabricated by lithographically defined aluminum electrodes on top of the nanofibers deposited on silicon dioxide grown thermally on a silicon substrate. The performance of a TiO_2 nanofiber humidity sensor was tested by AC and DC electrical measurements at 40–90% relative humidity. The response and the recovery time were 1 s and 4 s, respectively, between 40% and 90% relative humidity. The sensitivity of the TiO_2 humidity sensor in the range of 40–90% RH was 150 MΩ/%RH and 20 MΩ/%RH at 10 Hz and 100 Hz, respectively. The excellent sensing characteristics are attributed to the porous nature and the small diameter of the nanofibers.
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1. Introduction

In recent years, one-dimensional nanostructures, e.g., nanowires, nanotubes, and nanofibers, have been attracting much interest in physical, chemical, and biological sensor studies due to their unique properties such as small size and large surface-to-volume ratio [1–4]. Humidity sensors belong to chemical sensor category and have applications in semiconductor and automobile industries, pharmaceuticals, environmental control, food storage, and many other fields [5,6]. Among various materials, ceramic oxides (e.g., titanium dioxide) have also shown good sensing properties. Titanium dioxide (TiO_2) has also a variety of other applications in environmental cleaning and protection, photocatalysis, and solar cells [7]. Recently, a high-performance humidity sensor has been demonstrated using porous TiO_2 prepared by template-assisted sol–gel process [8]. Humidity sensing has also been reported using ZnO/TiO_2 double-layer nanofibers [9], TiO_2 nanotubes [10], ZnO/TiO_2 core shell nanorods [11], and TiO_2 films [12]. Mg^{2+} and Na^{+} doped rutile TiO_2 nanofibers have been used as antifogging humidity sensors as well [13].

In this work, we present a humidity-sensing device based on TiO_2 nanofibers prepared by electrospinning. Electrospinning is a simple, versatile, and relatively inexpensive technique for synthesis of nanofibers. Electrospinning has the advantage of synthesis of continuous, uniform nanofibers using different types of polymers and precursors [14,15]. Morphology of the nanofibers can be controlled well by viscosity, surface tension, and the density of net charges of the solution [14,15]. The nanofibers were prepared with an average diameter of ~150 nm and length of few hundred micrometers. The humidity sensor was tested at room temperature by varying the relative humidity from 40% to 90%. The TiO_2 nanofiber humidity sensor shows fast response and recovery time, and very good sensitivity. The sensitivity, response time and recovery time of the TiO_2 nanofiber sensor is comparable with previously reported TiO_2-based sensors discussed earlier. Further electrospinning is a simple technique to prepare nanofibers.

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2. Materials and methods

TiO_2 nanofibers were prepared by the electrospinning technique. The chemicals used in electrospinning (polyvinylpyrrolidone (PVP), titanium tetraisopropoxide, acetic acid, and ethanol) were purchased from Sigma–Aldrich. The chemicals were of analytical grade and used without further purification. The molecular weight of PVP was 1,300,000. Spin dope for electrospinning of TiO_2 nanofibers was prepared in the following manner. Solution 1 was prepared by adding 0.45 g of PVP in 7.5 ml of ethanol and stirred for 10 min. Then, solution 2 was prepared by mixing 1.5 ml of titanium tetraisopropoxide, 3 ml of ethanol and 3 ml of acetic acid in a glove box. Solution 2 was then poured dropwise in solution 1. The final solution was stirred for 1 h to get a homogenous solution of optimum viscosity.

Spin dope was then loaded in a plastic syringe with a stainless steel needle of 0.413-mm diameter. The copper collector plate was placed at distances of 7 cm, 8 cm, and 10 cm from the needle tip. High-voltage (10 kV) was supplied between anode (syringe) and cathode (copper plate). Composite (PVP/TiO_2) nanofibers were collected on an aluminum foil while the solvent evaporated during spinning. Uniform nanofibers with minimum beaded structure were obtained at an applied field of 1.25 kV/cm (i.e., when the distance between the needle and the collector was 8 cm). Pure TiO_2 nanofibers were obtained after heat treatment of as-spun PVP/TiO_2 composite nanofibers at 600 °C for 3 h in air. This resulted in removal of PVP from as-spun nanofibers. The nanofibers were characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), transmission electron microscopy (TEM), and X-ray diffraction (XRD).

To fabricate the humidity sensor, TiO_2 nanofibers were dissolved in iso-propanol by ultrasonic agitation. The nanofibers were then deposited on ~200-nm-thick SiO_2 grown thermally on the silicon substrate. Finally, aluminum electrodes were defined by lithography and thermal evaporation. Humidity-sensing devices with different separation between the aluminum electrodes were fabricated. Multiple nanofibers were present between the electrodes.

A home-made humidity measurement set-up was used for humidity measurements. Humidity-sensing devices were placed in a glass chamber. Probes for electrical measurements were fitted in the chamber. The DC and AC electrical measurements at different relative humidity (RH) were performed using a Keithley 2400 source meter and an Agilent E4980A LCR meter, respectively. Use of AC signal helps to avoid the polarization effects of adsorbed water [16]. However, in this case, the signal processing circuits are complicated. Therefore, sometimes, DC measurements are also used to evaluate the humidity sensors [16]. Saturated salt solutions were used to achieve required humidity levels. Saturated salt solutions of magnesium nitrate ($Mg(NO_3)_2$), sodium chloride (NaCl), and potassium chloride (KCl) were used to achieve 52%, 75%, and 86% RH, respectively at 24 °C [13]. Before AC and DC electrical measurements, each saturated salt solution was kept in the glass chamber for more than 24 h to attain the required RH. The RH values were confirmed by a commercial hygrometer before the electrical measurements.

3. Results and discussion

The XRD pattern of the TiO_2 nanofibers after heat treatment at 600 °C is shown in Fig. 1. It can be seen that anatase and rutile mixture phases are present. Fig. 2(a) shows the SEM image of the TiO_2 nanofibers after annealing at 600 °C. Fig. 2(b) shows the SEM image at a higher magnification. It can be seen that only the TiO_2 nanofibers are present. No other morphology such as beaded structure is observed. The nanofibers are very long (length in micrometers) and have diameters from ~30 nm to ~200 nm. EDS analysis of TiO_2 nanofibers shown in Fig. 2(c) reveals the presence of Ti and O in the atomic ratio of 1:2, indicating the formation of TiO_2. No other elements are present in the sample. This confirms that heat treatment at 600 °C removes PVP completely from the as-spun nanofibers. Fig. 3(a) shows a transmission electron microscope image of the TiO_2 nanofibers. The nanofibers are polycrystalline with average grain size of ~10 nm. The nanofibers have a porous structure. Lee et al. have reported that porosity depends on the calcination temperature [17]. They demonstrated that the nanofibers calcinated between 600 and 700 °C had porosity of 8% [17]. Because our nanofibers were prepared by the same technique and were calcined at 600 °C, we assume the nanofibers have similar porosity (not exactly 8%).

Fig. 3(b) shows the schematic diagram of the humidity-sensing device. Fig. 4(a) and (b) shows current voltage (*IV*) curves recorded at different relative humidity for devices A and B, respectively. The separation between two Al electrodes was ~200 μm for device A and 100 μm for device B. It is clear that the sensor responds to humidity change and current increases as the RH increases. This implies that the sensor offers low resistance at higher RH. These *IV* curves are nonlinear. Similar values of the current are observed for both devices, although the separation between the electrodes is different. The current flow in the nanofiber sensor depends on the distance between the

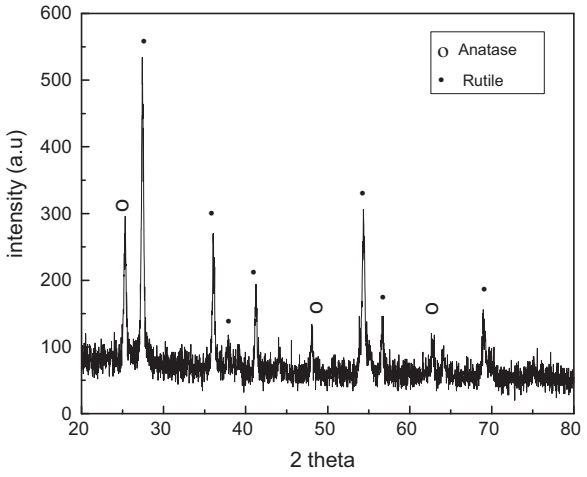

Fig. 1. X-ray diffraction pattern of the TiO_2 nanofibers.

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