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Rapid communication

Silica nanofibers based impedance type humidity detector prepared on glass substrate

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ABSTRACT

Impedance type relative humidity detector is fabricated by depositing electrospun silica nanofibers on glass substrate. The silica nanofibers with an average diameter ~150 nm and length ~100 μm were used. Thermogravimetric and differential scanning calorimetric analysis confirm that the accurate annealing temperature is 500 °C for complete removal of PVP. Humidity detecting devices were fabricated by defining titanium electrodes on top of the silica nanofibers. The performance of silica nanofibers humidity detectors was tested by AC electrical measurements at 40–90% relative humidity. The response and the recovery times were 5 s and 3 s, respectively, between 40% and 90% relative humidity. Contribution of dipoles, space charge polarization, relaxation of these dipoles and low frequency dispersion phenomenon were observed during impedance measurements.

Silica (SiO_2) and silica based nanomaterials are favored insulating materials for their peculiar and fascinating electrical applications such as in modern IC industries. It is an excellent electrical insulator and capable of forming a nearly perfect electrical interface with its substrate [1–3]. Moreover, especially due to its regular pore structure and its potential and demonstrated applications it has been extensively studied in many fields such as sensors, adsorbents, separation media and catalyst support [4–7]. Recently, silica based nanodielectric devices are being increasingly investigated due to their size and morphology dependent physicochemical properties and potential application superior to their bulk counterparts [8].

Among all the types of sensors, relative humidity (RH) sensor is one of the most commonly demanded due to need for control over humid environment in industry, laboratory work, processing of chemicals, food, pharmaceuticals, electronics, and textiles. Controlled humid environment is frequently required in the semiconductor manufacturing for the detection of trace moisture in many types of pure gases [9]. Different techniques for different nanostructures such as particles, belts, sheets, combs, rings, rods and tubes have been attracted much attention for fabrication of RH sensors [10–12]. As the mechanism of RH sensors is mainly associated with adsorption and desorption processes, so the surface area of the sensing materials becomes an important factor to determine the sensing properties [13]. Here, we use the one dimensional nanostructure for RH sensor synthesized by relative simple and low cost electrospinning technique.

Among solid-state sensors, those based on the measurement of electrical properties, such as resistance or capacitor is easier to implement and are widely used in many applications [14,15]. Moreover, the capacitive and resistive RH sensors can directly measure the capacitance and impedance changes with fast response, high linearity, low hysteresis, and excellent long term stability [16]. In resistive type RH sensors, electrons, ions, protons and dipoles are responsible for conducting electricity in different RH ranges [17]. In some recently published articles [18–22], dielectric properties of silica based hybrid nanostructures and thin films have been investigated in which capacitance and dielectric constant as a function of frequency is studied but the effect of RH on dielectric response of SiO_2 nanofibers is rarely investigated.

Focus of this research is to build an RH detector based on silica nanofibers prepared by a reliable and low cost electrospinning technique. The RH sensing properties of SiO_2 detector exhibits

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a remarkably improved performance due to its large surface to volume ratio, resulting in high sensitivity, fast response, small hysteresis and long term stability.

Polyvinylpyrrolidone (PVP, M*w* ~ 13, 00000), tetraethoxysilane (TEOS) > 98%, ethanol (C_2H_5OH > 98%) and acetic acid (CH_3COOH > 98%) were used as starting materials. The reagents were of analytical grade and were purchased from Sigma Aldrich. The chemicals were used without any further purification. To synthesize SiO_2 nanofibers, 13% PVP/ethanol and 13% TEOS/acetic acid solutions were prepared. The two solutions were then mixed (the volume ratio of the two solutions i.e. PVP/ethanol: TEOS/acetic acid = 15:1) thoroughly for 3 h. The final solution was then loaded into a syringe and electrospinning was carried out at ambient conditions using a 0.511 mm diameter stainless steel needle. The collector plate was placed at 7 cm from the needle. A high voltage 10 kV was applied between the needle and collector plate. As spun PVP/SiO_2 nanofibers were collected on aluminum foil place on collector plate. The composite PVP/SiO_2 nanofibers were left in the air for 24 h for hydrolysis of TEOS. Subsequently, PVP/SiO_2 nanofibers were annealed at 500 °C for 6 h in air to obtain pure SiO_2 nanofibers.

To fabricate the humidity detector 7740 glass having excellent insulating properties was used as a substrate. 5 g SiO_2 nanofibers were mixed with 100 ml isopropanol using ultrasonic bath. A drop of the isopropanol containing SiO_2 nanofibers was spin coated onto glass substrate at 2000 rpm. This led to formation of nanofibers mat on the glass substrate. Then 80 nm Ti electrodes were evaporated using a shadow mask. 20 nm Cr was evaporated before the evaporation of Ti to improve adhesion with nanofibers. The separation between the electrodes was ~90 μm Fig. 1 (a) shows the schematic diagram of the final device.

Fig. 1 (b) shows the scanning electron microscope (SEM) image of pure SiO_2 nanofibers after the removal of PVP, annealed at 500 °C for 6 h. The nanofibers are very long having lengths ~100 μm with diameters ~150–200 nm. The nanofibers are porous as can be seen from the high magnification SEM image shown in Fig. 1 (c). The porous nature of nanofibers is useful in humidity detection as it increases the surface area. Fig. 1 (d) shows the energy dispersive spectrum of SiO_2 nanofibers. The presence of atomic % of Si and O in the sample indicates formation of SiO_2 and complete removal of PVP.

Thermogravimetric and differential scanning calorimetry scans of SiO_2 nanofibers are shown in the Fig. 2. These scans were obtained using TGA Q500 and DSC Q100 under nitrogen flow (10 mL/min). The sample size and heating rate was 1.950 mg and 10 °C/min, respectively. It can be seen that there is an overall weight loss ~17% between 20 °C and 500 °C. This is attributed to removal of moisture absorbed and any remaining solvent. With further increase in temperature, no weight loss occurs indicating the thermal stability of SiO_2 nanofibers. Therefore thermal analysis of SiO_2 nanofibers confirms that correct annealing temperature is 500 °C for complete removal of PVP [23,24]. The endothermic peak in differential scanning calorimetric scan at ~77 °C corresponds to the removal of absorbed water in the pores of the surface of SiO_2 nanofibers. These pores are of small radius. It is difficult to remove adsorbed water from pores. Therefore strong endothermic peak appears in Fig. 2 [25].

SiO_2 nanofibers humidity detector was tested from 40 to 90% RH in a climate chamber (Blue M electric, Japan). The impedance

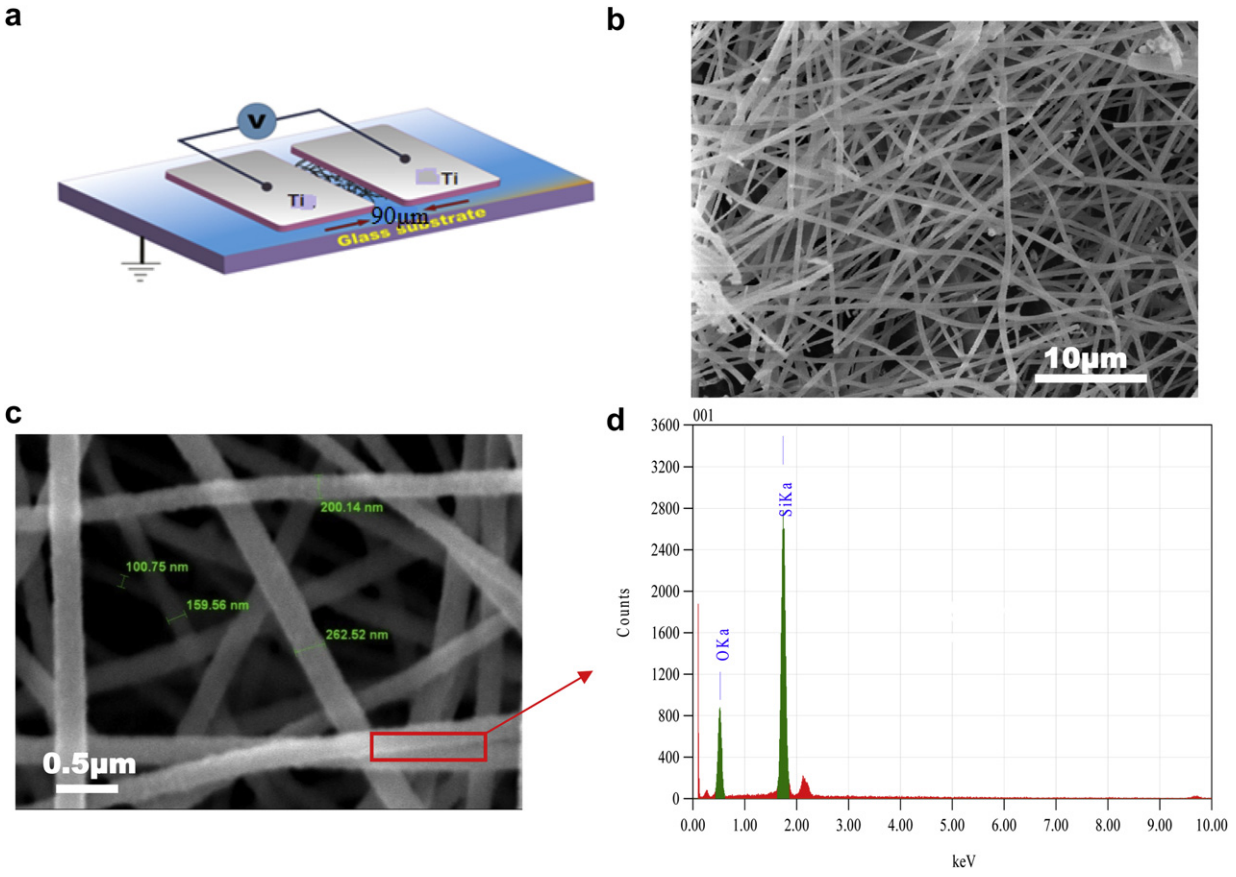

Fig. 1. (a) Schematic diagram of impedance type humidity detector, SEM images of SiO_2 nanofibers heat treated at 500 °C for 6 h (b) low resolution (c) high resolution (d) EDS spectrum of selected area.