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Ceramics International 39 (2013) 457–462

CERAMICS INTERNATIONAL

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Excellent humidity sensing properties of cadmium titanate nanofibers

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Received 24 February 2012; received in revised form 30 May 2012; accepted 15 June 2012
Available online 23 June 2012

Abstract

We report humidity sensing characteristics of $CdTiO_3$ nanofibers prepared by electrospinning. The nanofibers were porous having an average diameter and length of ~50–200 nm and ~100 μm, respectively. The nanofiber humidity sensor was fabricated by defining aluminum electrodes using photolithography on top of the nanofibers deposited on glass substrate. The performance of the $CdTiO_3$ nanofiber humidity sensor was evaluated by AC electrical characterization from 40% to 90% relative humidity at 25 °C. The frequency of the AC signal was varied from 10^{-1} to 10^6 Hz. Fast response time and recovery time of 4 s and 6 s were observed, respectively. The sensor was highly sensitive and exhibited a reversible response with small hysteresis of less than 7%. Long term stability of the sensor was confirmed during 30 day test. The excellent sensing characteristics prove that the $CdTiO_3$ nanofibers are potential candidate for use in high performance humidity sensors.

Keywords: Cadmium titanate; Nanofibers; Humidity sensor; Response time

1. Introduction

Humidity sensors are of great interest to research community due to their vast application in daily life, industry and research [1–4]. The desirable sensing characteristics of a humidity sensor have short response and recovery time, excellent reproducibility for a long time, low temperature and low cost, high sensitivity and good chemical and thermal stability. To achieve this, various materials [2,5–8] in different forms e.g. nanoparticles [9], nanofibers [7], and thin films [10] have been exploited. However humidity sensors based on one-dimensional nanostructures such as nanowires and nanofibers have shown greater performance due to large surface to volume ratio [9]. Therefore there are pressing needs for preparation of new materials for use in sensors. Here we report a new one-dimensional nanostructure: $CdTiO_3$ nanofibers to be used in humidity sensors. The $CdTiO_3$ nanofibers are prepared by a simple yet versatile technique electrospinning. Electrospinning provides a convenient and facile route for the formation of long nanofibers with uniform diameters. The morphology of the nanofibers can easily be tailored by adjusting experimental parameters (e.g. applied voltage, distance between needle and collector, etc.) and the parameters of the feed solution (e.g. viscosity, polymer concentration, etc.). The details of the preparation of $CdTiO_3$ nanofibers can be found elsewhere [11].

Humidity sensors based on nanofibers of various titanates have been investigated. Wang et al. reported highly sensitive $BaTiO_3$ electrospun nanofibers and studied DC humidity sensing properties of fabricated device [12]. He et al. reported an advanced impedance type humidity sensor based on electrospun $BaTiO_3$ nanofibers [7]. A fast, simple and reliable humidity sensor based on KCl-doped SnO_2 electrospun nanofibers has been reported by Song et al. [13]. Li et al. reported highly sensitive and stable

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0272-8842/$36.00
http://dx.doi.org/10.1016/j.ceramint.2012.06.048

humidity nanosensor based on LiCl doped TiO_2 electrospun nanofibers [14]. However there are no studies on humidity sensing characteristics of $CdTiO_3$ nanofibers. Therefore in this work we present first time studies on humidity sensing characteristics of $CdTiO_3$ nanofibers. The sensing device of $CdTiO_3$ nanofibers prepared by this method exhibits remarkably improved performance due to its porous structure and small diameter of nanofibers.

2. Materials and methods

2.1. Sample preparation

Cadmium titanate nanofibers were synthesized by the electrospinning technique. Cadmium acetate ($Cd(CH_3COO)_2 \cdot 2H_2O > 98\%$), titanium tetraisopropoxide ($Ti((CH_3)_2CHO)_4 > 99\%$), acetic acid ($CH_3COOH > 98\%$), ethanol ($C_2H_5OH > 98\%$) and polyvinyl-pyrrolidone (PVP) (M_W= 13,00,000) were purchased from Sigma Aldrich. All chemicals were used without any further purification and were of analytical grade. Two grams of cadmium acetate was mixed with 4 mL of acetic acid at room temperature in a glove box followed by dropwise addition of titanium tetraisopropoxide. The second solution was prepared by adding 0.5 g of PVP in 4 mL ethanol under stirring for 15 min. These two solutions were then mixed and vigorously stirred for 3 h in a glove box to get a homogeneous solution of optimum viscosity.

The resulting solution was loaded in a syringe with a 24-gauge needle. The needle and collector plates were connected to the positive and negative terminal of high voltage supply of 10 kV, respectively. The nanofibers were collected on an aluminum foil placed on the collector plate. The distance between the tip of the needle and collector plate was 7 cm. Uniform nanofibers were obtained at an applied field of 1.42 kV/cm. The whole process of electrospinning was conducted in an open atmosphere. The as-spun nanofibers were dried in an oven at 80 °C for 3 h. Finally, PVP was evaporated to obtain pure $CdTiO_3$ nanofibers by heat treatment of the nanofibers in air at 600 °C for 3 h with a heating rate of 5 °C/min.

2.2. Device fabrication of $CdTiO_3$ nanofibers

To study the humidity sensing properties, a suspension of the $CdTiO_3$ nanofibers in isopropanol was prepared with weight ratio of 1:100. In order to achieve the uniform suspended solution, the suspension was placed in the ultrasonic bath for 30 min. Nanofibers were then deposited on the pre-cleaned glass substrate using the spin coater. Aluminum electrical contacts were deposited using thermal evaporation and photolithography. The separation between the two electrical contacts was 25 μm. More than one nanofiber was present between the aluminum contacts.

2.3. Measurements

X-ray diffraction (XRD) analysis was conducted on a Brag-Brentano X-ray diffractometer with Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å). Field emission scanning electron microscopy (SEM) images were obtained by using a JSM-6490LA, Japan. The characteristic curves of RH response were measured by dielectric spectrometer (broad band dielectric spectrometer novo control, Germany). The AC signal amplitude used in our studies was 1 V while the frequency was varied from 10^{-1} Hz to 10^6 Hz. The WINDETA software was used for output data, which is fully automated by interfacing the dielectric spectrometer with a computer. A well controlled RH environment was achieved by a climate chamber (Blue M electric, Japan). The chamber had two gas inlets, one for the dry air and the other for water vapors. During measurements, RH in the chamber was controlled by an automatic electronic controller. The air chamber was also controlled by heating/cooling system. The complete RH measurements setup was maintained at ~25 °C in a clean-room environment.

3. Results and discussion

3.1. Structural characterization of $CdTiO_3$ nanofibers

Fig. 1(a) shows XRD pattern of the $CdTiO_3$ nanofibers. Diffraction peaks in the XRD pattern can be indexed by ilmenite phase of $CdTiO_3$ with rhombohedral structure. The XRD pattern agrees with the JCPDS Card no. 29-0277 and lattice parameters determined from Fig. 1(a) are $a = 5.29$ Å and $c = 14.874$ Å. Fig. 1(b) shows the scanning electron microscope (SEM) image of $CdTiO_3$ nanofibers annealed at 600 °C. The nanofibers have an average diameter ~50–200 nm. The length of the nanofibers is ~100 μm. Fig. 2(a) shows the high magnification image of the nanofibers. It can be clearly seen that the nanofibers are porous in nature. The bright field transmission electron microscope image shown in Fig. 2(b) confirms the porous nature of the nanofibers. The porosity is useful for sensing humidity as it increases the effective area of the $CdTiO_3$ nanofibers for the adsorption and desorption processes due to possible in and out diffusions of humidity molecules through the pores and increase the sensitivity [9,15].

3.2. Humidity sensing measurements

Fig. 3(a) shows the schematic diagram of the $CdTiO_3$ nanofiber humidity sensing device. Fig. 3(b) shows top view of the nanofiber humidity sensing device recorded by SEM. More than one nanofibers are present between the metal contacts. The $CdTiO_3$ nanofiber humidity sensor was tested using impedance measurements at ambient temperature and relative humidity was varied from 40% to 90%. However, different atmospheres can also be used [16]. Here frequency was varied from 10^{-1} Hz to 10^6 Hz.

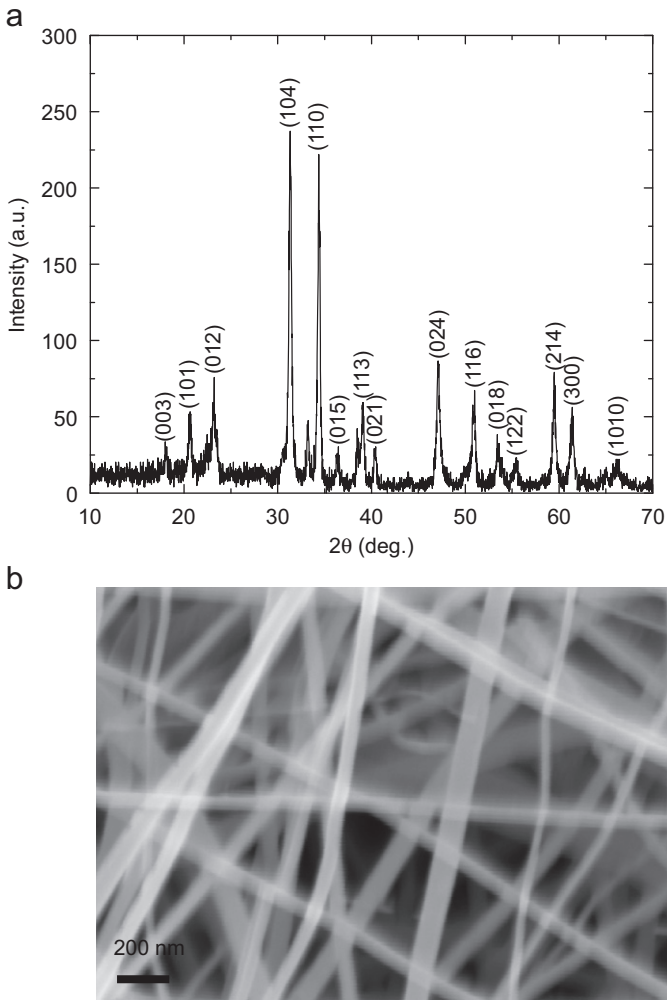

Fig. 1. (a) XRD pattern of $CdTiO_3$ nanofibers after annealing at 600 °C for 3 h and (b) SEM image of $CdTiO_3$ nanofibers.

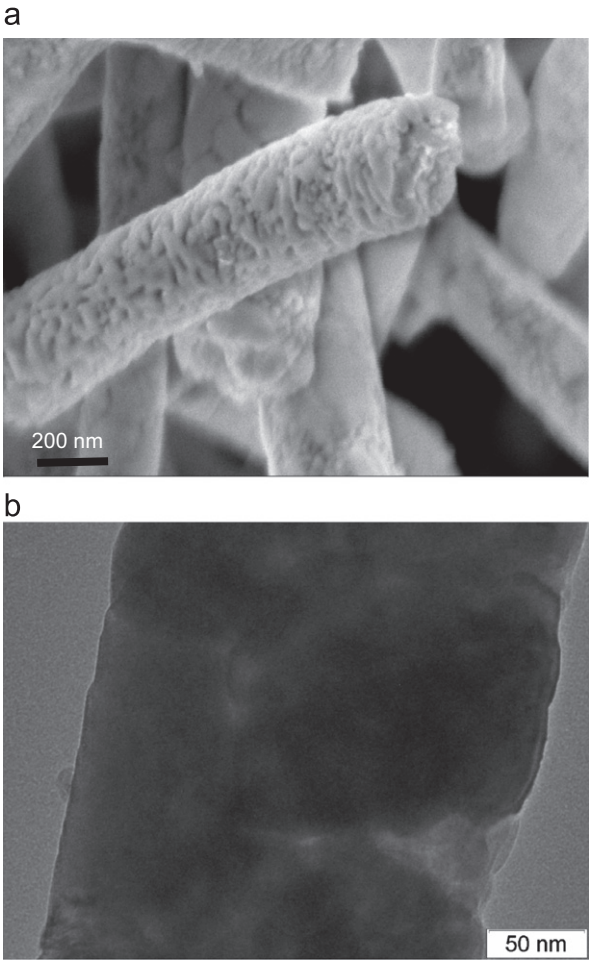

Fig. 2. (a) SEM image of $CdTiO_3$ nanofibers at high magnification and (b) TEM image at high magnification.

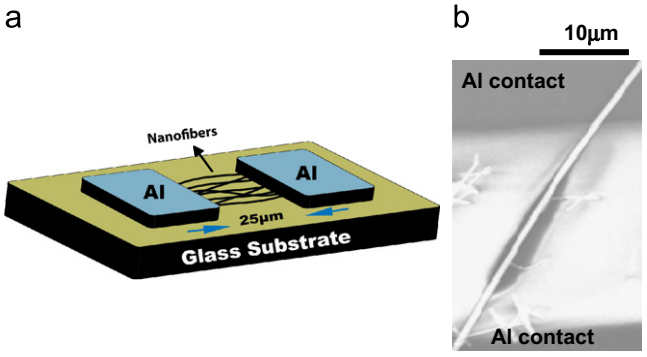

Fig. 3. (a) Schematic diagram and (b) SEM image of nanofiber humidity sensor.

Fig. 4(a) shows the effect of relative humidity on the impedance at three different frequencies, 10^5 Hz, 10^3 Hz and 10^2 Hz. The impedance of $CdTiO_3$ humidity sensor decreases as RH increases. The value of impedance decreases from $7.4 \times 10^5\ \Omega$ (40% RH) to $6.4 \times 10^5\ \Omega$ (90% RH) at 10^5 Hz, $6.7 \times 10^7\ \Omega$ (40% RH) to $1.3 \times 10^7\ \Omega$ (90% RH) at 10^3 Hz and $6.0 \times 10^8\ \Omega$ (40% RH) to $3.9 \times 10^7\ \Omega$ (90% RH) at 10^2 Hz. The impedance of the nanofiber humidity device has strong dependence on the humidity at lower frequencies as compared to impedance change at higher frequencies. The sensor has almost linear response at all frequencies.

The relationship between capacitance of the nanofiber humidity sensor and frequency at 40–90% RH is shown in Fig. 4(b). It is evident that capacitance increases with an increase in RH at low frequencies. At high frequencies ($> 10^3$ Hz), capacitance has low values and it is almost independent of the RH. The inset of Fig. 4(b) shows the frequency dependence of the capacitance of nanofiber humidity sensor. The change in capacitance is due to variation of dielectric constant of the nanofiber sensor in presence of water. It has been demonstrated that [9] capacitance is directly proportional to the leak conductance and inversely proportional to the frequency. However leak conductance increases with increase in RH. This indicates that the capacitance of the nanofiber sensor increases with RH, depending on frequency. At high frequencies the speed of polarization of water molecules

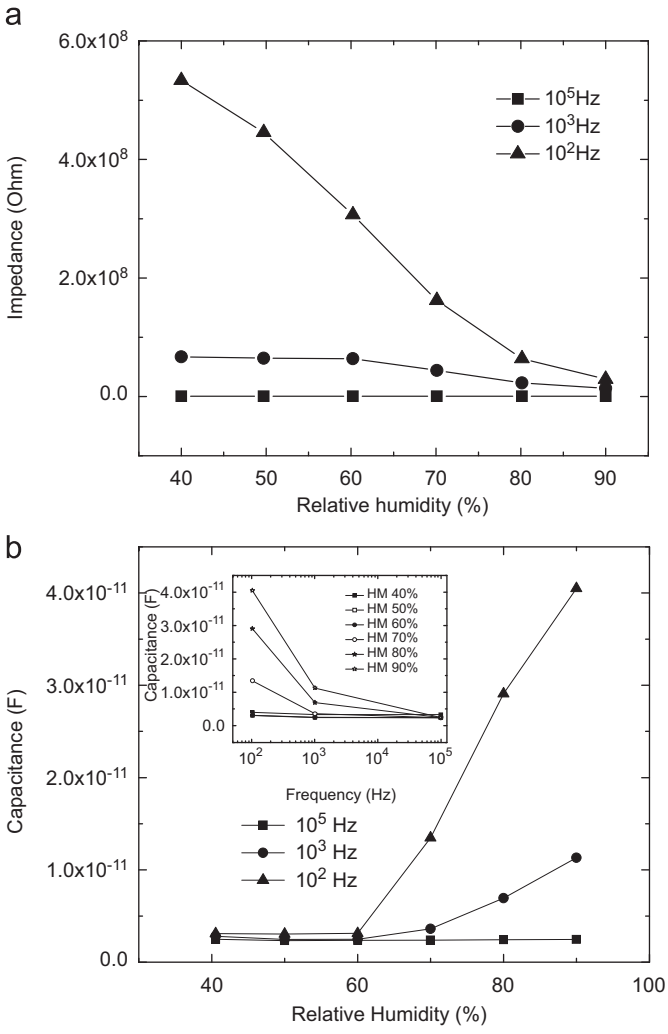

Fig. 4. (a) Impedance versus RH measured at various frequencies and (b) capacitance versus frequency at various RH for nanofiber sensor measured at 1 V (inset: corresponding capacitance–frequency curve).

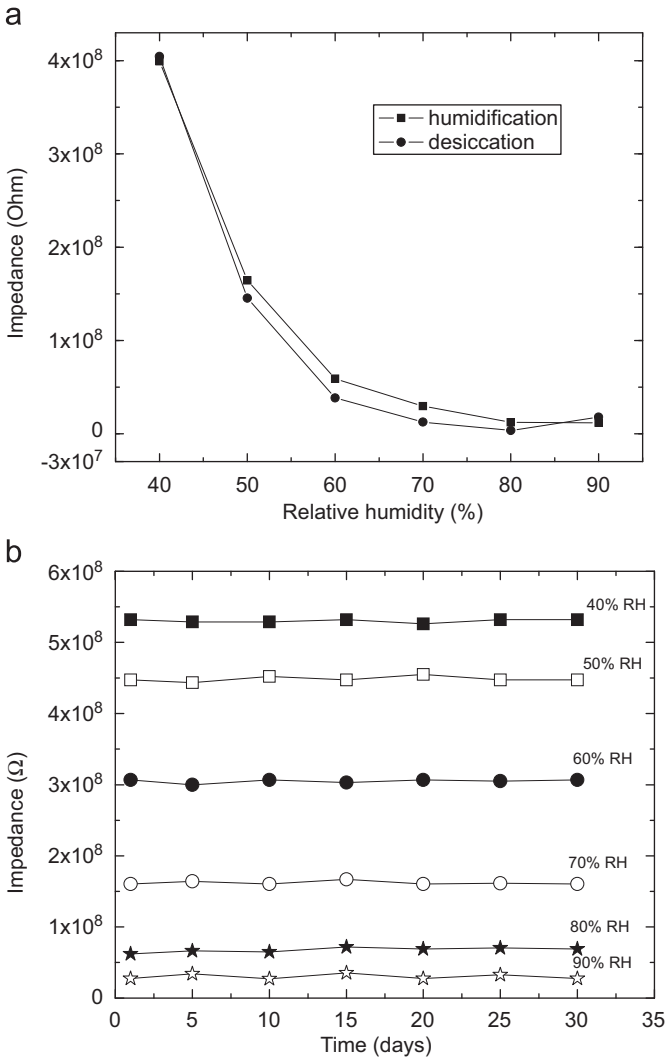

Fig. 5. (a) Humidity response of the sensor based on $CdTiO_3$ nanofibers during humidification and desiccation process and (b) long term stability property of sensor over 30 days measured at 10^2 Hz and 1 V.

is slower as compared to the rapid changes in the direction of the electric field. Therefore at high frequencies the capacitance is small and independent of frequency [9,17].

The sensitivity of the nanofiber sensor can be calculated from Fig. 4(a) using the following expression [18]:

$$\text{Senstivity} = \frac{\Delta(\text{impedance})}{\Delta\%\text{RH}} \quad (1)$$

The sensitivity in the range of 40–90% RH at 10^2 Hz is found to be 10.01 MΩ/% RH.

The humidity hysteresis is another important figure of merit of a humidity sensor. The humidity hysteresis is defined as the maximum difference between the humidification and desiccation curve. The humidity hysteresis characteristics of $CdTiO_3$ nanofiber sensor are shown in Fig. 5(a). The maximum hysteresis of ~7% is observed in our devices at 100 Hz, indicating good reliability of the sensor.

To test the long term stability, the nanofiber sensor was kept for 30 days in air at room temperature, and the impedance measurements were performed after every 5 days at different RH values. The frequency and AC voltage were kept constant during these measurements (AC voltage 1 V and $f = 100$ Hz). Fig. 5(b) shows the results of these measurements. Almost no change in the impedance of the device is observed over 30 days, confirming the good stability of the presented RH sensor.

The increase in conductivity of $CdTiO_3$ nanofibers with RH could be understood by the Grotthuss mechanism which relates this behavior with the tunneling of proton from one water molecule to the neighboring water molecule [19]. At lower humidity, water molecule is chemically adsorbed (chemisorption) on the activated site on the surface of $CdTiO_3$ nanofiber. At this stage two hydroxyls are formed per water molecule by a dissociative mechanism. With further increase in humidity, the physical adsorption (physisorption) of water molecules occurs. Physisorption is a multilayer phenomenon. The first layer of physically adsorbed water is due to double hydrogen bonding of a single molecule

and this layer is immobile. The humidity molecules in subsequent physisorbed layers form single bond and thus protons may be available for conduction. With increase in humidity, the physisorbed layers increase and more protons are available for conduction.

We have performed our experiment under ambient conditions and at this temperature ionic conduction is dominant due to moisture content in the pores of the sensing material. However, it is reported that with increasing temperature, moisture content reduces so the conduction process is dominant by electronic conduction instead of ionic conduction [20].

The response time measurements are performed by recording the impedance of the nanofiber sensor when relative humidity was changed between 40% and 90% keeping the AC voltage=1 V and frequency f=100 Hz. The response–recovery curve of the nanofiber humidity sensor is shown in Fig. 6. The response time of $CdTiO_3$ nanofiber sensor is found to be as small as 4 s. The recovery time is ~6 s.

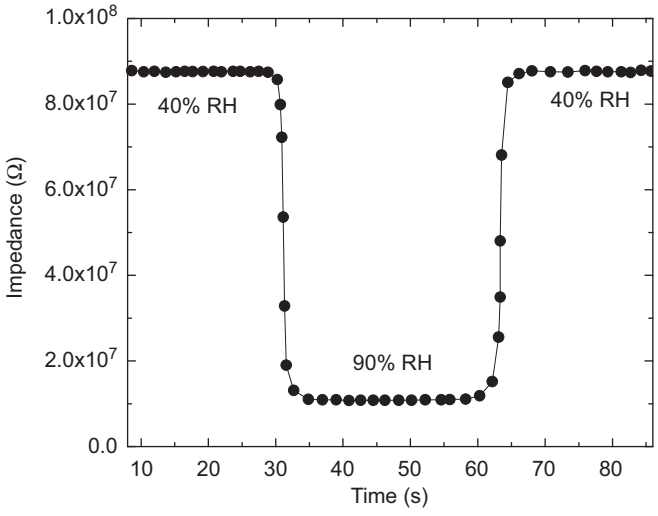

Fig. 6. Response and recovery curve of $CdTiO_3$ nanofibers measured at 10^2 Hz.

Table 1 compares the performance of our nanofiber sensor with a few recently reported titanate and metal oxide based humidity sensors. The hysteresis, the response time and recovery time of the $CdTiO_3$ nanofiber sensor are better or comparable with other sensors discussed in Table 1. However, the sensitivity of the $CdTiO_3$ nanofiber humidity sensor is better than the sensors (where such data is available) presented in Table 1. Sensitivity for $BaTiO_3$ nanofibers (Fig. 4 in Ref. [7]), $BaTiO_3$/polystyrene film (Fig. 6 in Ref. [21]), $BaTiO_3$ thin film (Fig. 4 in Ref. [22]), ZnO/TiO_2 double-layer nanofibers (Fig. 5 in Ref. [23]), and porous TiO_2 (Fig. 6 in Ref. [9]), ZnO/TiO_2 composite nanofibers (Fig. 3 in Ref. [24]), and $Bi_{0.5}K_{0.5}TiO_3$ powder (Fig. 4 in Ref. [25]), were calculated by the authors using Eq. (1). Further the nanofibers are easier to prepare. The best performance of the $CdTiO_3$ nanofibers is due to the small diameter and porous nature of the nanofibers. Therefore $CdTiO_3$ nanofibers are good candidate for use in humidity sensing devices. The sensing properties of our nanofibers can be further improved by reducing the diameter of the nanofibers which can easily be reduced by adjusting the experimental parameters during electrospinning.

4. Conclusion

In summary, we have successfully fabricated impedance type humidity sensor based on porous $CdTiO_3$ nanofibers. The nanofibers having diameter of ~50–200 nm and ~100 μm long, were prepared by electrospinning. The humidity sensor was fabricated using standard microfabrication techniques. Aluminum metal was used to form metal contacts to the nanofibers. The $CdTiO_3$ nanofibers exhibit good humidity sensing characteristics including high senstivity, small hysteresis (< 7%), fast response time (4 s), fast recovery time (6 s)and stable operation over long

Table 1
Comparison of the $CdTiO_3$ nanofiber humidity sensor with other humidity sensors.

Material	Humidity range (%)	Response time (s)	Recovery time (s)	Sensitivity at 100 Hz (MΩ/% RH)	Hysteresis (% RH)	Reference
$BaTiO_3$ nanofibers	11–95	5	4	0.59	5	[26]
$BaTiO_3$/polystyrene sulfonic sodium	7–98	50	120	0.83	8	[21]
$BaTiO_3$ thin film	30–90	30	40	0.16	–	[22]
Double layered ZnO–TiO2 nanofibers	11–95	11	7	0.65	1.5	[23]
Porous TiO_2	11–95	5	8	0.50	< 1	[9]
$Ba_{0.7}Sr_{0.3}$ TiO_3	11–95	42	42	–	3	[27]
ZnO/TiO_2 composite nanofibers	10–90	4	12	0.40	2	[24]
Nano $BaTiO_3$-polymer RMX	7–98	15	120	0.55	–	[28]
$Bi_{0.5}K_{0.5}TiO_3$ powder	11–95	12	25	0.57	3	[25]
$ZrTiO_4$	32–85	–	–	–	–	[5]
$CdTiO_3$ (thin film)	4–87	–	–	–	–	[29]
$CdTiO_3$ (present work)	40–90	4	6	10.01	< 7	–

period. These good characteristics are attributed to small diameter and porous nature of the nanofibers.

Acknowledgments

We acknowledge the financial support of the Higher Education Commission (HEC), Pakistan.

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