Sensors and Actuators B 194 (2014) 260–268

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

Rapid synthesis of cerium oxide nanoparticles with superior humidity-sensing performance

Sharada Thakur, Pradip Patil*

Department of Physics, North Maharashtra University, Jalgaon 425001, India

ARTICLE INFO

Article history:
Received 22 October 2013
Received in revised form 6 December 2013
Accepted 16 December 2013
Available online 24 December 2013

Keywords:
Cerium oxide nanoparticles
Microwave synthesis
Humidity sensor
Nyquist impedance plots
X-ray diffraction
Transmission electron microscopy

ABSTRACT

This paper reports a simple and rapid microwave-assisted method for synthesizing cerium oxide (CeO_2) nanoparticles for the fabrication of high-performance humidity sensors. The humidity-sensing investigation reveals that the sensor based on CeO_2 nanoparticles exhibits a high and linear response within the entire relative humidity (RH) range of 11–97% at an operating frequency of 60 Hz. The corresponding impedance changes by approximately three orders of magnitude within the entire humidity range from 11% to 97%. The response and recovery times are approximately 3 and 16 s, respectively. Additionally, the sensor exhibits a rapid and reversible response characterized by a very small hysteresis (~1%RH), excellent repeatability, long term stability and a broad range of operation (11–97%RH). The Nyquist impedance plots of the sensor at different RHs were used to elucidate the sensor's humidity-sensing mechanism via an electrical equivalent circuit. The experimental results provide a possible method for the rapid synthesis and fabrication of high-performance humidity sensors based on CeO_2 nanoparticles.

© 2013 Elsevier B.V. All rights reserved.

1. Introduction

Over the last decade, considerable efforts have been made to develop suitable humidity-sensitive materials for the fabrication of high-performance humidity sensors [1–4]. To fabricate a sensor that is suitable for practical use, a high response value, a linear response, a fast response and recovery behavior, a small hysteresis, good reproducibility, a wide range of relative humidity detection, long-term stability and low cost are required. The performance of a humidity sensor mainly depends on the properties of the sensing materials. Metal oxides [5–7], polymers [8,9] and inorganic/organic hybrids [10–12] have been widely investigated as sensing materials for the fabrication of humidity sensors. Among the various sensing materials, metal oxides have been widely investigated due to their unique properties such as chemical and physical stability, high mechanical strength and a wide operating temperature range [5–7].

Recently, the nanostructures of metal oxides have received considerable interest in the fabrication of sensors to detect humidity and gases due to the large surface-to-volume ratio, high surface activity and effective electron transport of these oxides [13–19]. A wide range of nanostructured metal oxides such as SnO_2 nanowires [13], ZrO_2 nanorods [14], ZnO nanorods [15], Al_2O_3 nanowires [16], TiO_2 nanotubes [17], $BaTiO_3$ nanofibers [18] and $ZnSnO_3$ nanocubes [19] have been investigated as humidity-sensing materials during the past few years.

In the context of humidity sensors, CeO_2 is less studied but is a potential ceramic material for sensing applications due to the material's good response to humidity [20]. Recently, hydrothermally synthesized CeO_2 nanowires were used for humidity sensing by Fu et al. [20]. The CeO_2 nanowires indicate an exponential decrease of the wire's resistance with an increase in the RH, and both the response and the recovery times were found to be ~3 s. However, the synthesis method of CeO_2 nanowires is time-consuming. The humidity-sensing properties of the Ba-doped CeO_2 nanowires [21] and Mn-doped CeO_2 nanorods [22] synthesized by using a facile composite-hydroxide-mediated (CHM) route have been recently studied. However, the other humidity-sensing characteristics such as the humidity response, the response and recovery times, the hysteresis and the repeatability have not been investigated. A number of methods have been used to synthesize nanostructured CeO_2 including sol–gel [23], hydrothermal [24], homogeneous precipitation [25], reverse micelles [26], flame spray pyrolysis [27] and sonochemical methods [28]. However, some of these methods require long processing times, and usually, the ceria precursor formed during precipitation is calcined to obtain the product.

In this paper, we report the synthesis of CeO_2 nanoparticles by a simple, cost-effective and rapid microwave-assisted method and a study of the humidity-sensing characteristics of a humidity sensor based on CeO_2 nanoparticles. We have determined the response time, the recovery time and the humidity response to understand these sensor's true potential as alternative humidity sensors. The

* Corresponding author. Tel.: +91 2572257474.
E-mail address: pnmu@yahoo.co.in (P. Patil).

0925-4005/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.snb.2013.12.067

stability and repeatability of the response have also been studied. The obtained experimental results provide a possible method for the rapid synthesis and fabrication of high performance humidity sensors based on CeO_2 nanoparticles.

2. Experimental

2.1. Synthesis of CeO_2 nanoparticles

The synthesis of CeO_2 nanoparticles (NPs) was performed by employing a microwave-assisted method using analytical-grade ammonium cerium(IV) nitrate and propylene glycol without further purification. The ammonium cerium(IV) nitrate was used as the source of cerium, and propylene glycol was used as a stabilizing agent. Microwave irradiation was performed with a domestic microwave oven (CQ138S, Samsung, Japan) operated at a microwave frequency of 2.45 GHz with a maximum power of 800 W. In a typical experiment, 0.1 M each of ammonium cerium(IV) nitrate and propylene glycol were dissolved in double-distilled water and stirred continuously for 1 h at room temperature (25 °C). The appropriate amount of ammonia was added drop-wise to this solution with continuous stirring until the final pH value of approximately 10 was achieved in the solution. The resulting pale-yellow-colored precipitate was filtered, washed several times with double-distilled water and alcohol, and subjected to microwave irradiation at 600 W microwave power for 10 min. After irradiation, the yellow-colored precipitate was harvested by centrifugation, washed several times using double-distilled water and ethanol, and then dried in an oven at 90 °C overnight to obtain the end-product for further characterization.

2.2. Characterization

The structural analysis of the as-synthesized CeO_2 NPs was performed using an X-ray diffractometer (XRD, D8 Advance, Bruker AXS) with Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å). The surface morphological study was performed using a transmission electron microscope (TEM, 1200 EX, JEOL, Japan). The Fourier transform infrared (FTIR) spectroscopy analysis was performed using a FTIR spectrometer (FTIR, IMPACT 420 DSP, Nicolet) by the conventional KBr method in the spectral range 4000–400 cm^{-1}.

2.3. Sensor fabrication

In the present work, the sensor consisting of an interdigitated electrode (IDE) and a layer of CeO_2 NPs coated on top as a humidity-sensing material was fabricated. The size of the entire device is 23 mm × 15 mm and the typical dimensions of the sensing area of the CeO_2 NPs are 20 mm × 15 mm. The IDE consists of five pairs of Cu tracks screen-printed onto an epoxy glass substrate (25 mm × 20 mm). The width of a Cu track and the gap between two successive tracks are each 1 mm. Prior to use, the IDE-epoxy glass substrates were cleaned by an ultrasonic treatment in acetone, then rinsed thoroughly with double-distilled water and dried in vacuum. The as-synthesized CeO_2 NPs powder was mixed with double-distilled water in a weight ratio of 100:25 to form a paste. The paste was then spin-coated using the Spin Coater (SPN 2000, Milman Thin Film Systems, Pvt. Ltd., Pune, India) onto the IDE-epoxy glass substrates at 2000 rpm for 12 s to form CeO_2 films. After spin coating, the CeO_2 films were dried at 80 °C for 12 h and used as sensing elements to evaluate the humidity-sensing characteristics. The schematic diagram of a fabricated humidity sensor is shown in Fig. 1.

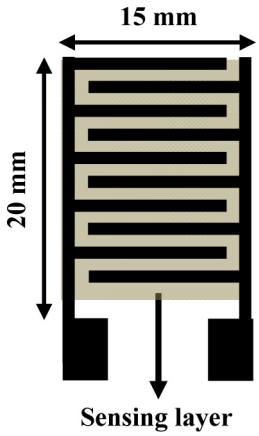

Fig. 1. Schematic diagram of a fabricated humidity sensor.

2.4. Humidity-sensing measurements

The different RH levels were generated by the different saturated salt solutions in closed bottles at room temperature. The bottles were made of glass with heights of 19 cm and diameters of 6 cm. The six different standard saturated aqueous salt solutions of LiCl (11 ± 0.30%RH), $MgCl_2$ (33 ± 0.14%RH), K_2CO_3 (43 ± 0.20%RH), NaCl (75 ± 0.15%RH), KCl (85 ± 0.24%RH) and K_2SO_4 (97 ± 0.16%RH) were used to act as humidity sources. The saturated salt solutions were placed in the bottles for 12 h to ensure that the air in the bottles reached equilibrium states. The CeO_2 film was placed successively into the bottles with different RH levels at room temperature, and the impedance of the film was measured as a function of RH. A humidity probe (Model 6517-RH Humidity probe, Keithley Instruments, USA) was also placed into the bottles along with the CeO_2 film to monitor the RH during the measurement. The impedance of the CeO_2 film was measured as a function of RH using a simple two-probe configuration with a LCR Meter 4300 (Wayne Kerr Electronics, USA) controlled by the test software supplied by Biotronic systems, Mumbai, India. The voltage applied was AC 1 V, and the frequency was varied from 60 Hz to 1 kHz.

The Nyquist impedance plots were also recorded using a Solartron impedance gain phase analyzer (SI 1260, Solartron, U.K.) in a two-point configuration in the relative humidity range 11% to 97% and in the frequency range 40 Hz to 100 kHz with an alternating-current excitation potential of 10 mV. The analysis of the impedance plots was performed by the fitting the experimental results to an electrical equivalent circuit with the Z-View software from Scribner Associates [29]. The quality of the fit to the equivalent circuit was judged first by the x^2 value and second by the limitation of the relative error in the value of each element in the equivalent circuit to 5%.

3. Results and discussion

3.1. XRD analysis

The XRD pattern of the as-synthesized product shown in Fig. 2(a) is in good agreement with that of the pure CeO_2 crystalline phase (JCPDS No.: 81-0792). All of the diffraction peaks can be indexed to CeO_2 with a face-centered cubic structure. No other peaks were observed, indicating that no impurities were present and confirming that the adopted synthesis method gives pure CeO_2 NPs. The average crystallite size was calculated by fitting the [1 1 1] diffraction peak ($2\theta = 27.76°$) width using a Gaussian function and

Download English Version:

https://daneshyari.com/en/article/742879

Download Persian Version:

https://daneshyari.com/article/742879

Daneshyari.com