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Characterization and humidity sensing properties of $Bi_{0.5}Na_{0.5}TiO_3$–$Bi_{0.5}K_{0.5}TiO_3$ powder synthesized by metal-organic decomposition

Yong Zhang[a], Xuejun Zheng[a,*], Tong Zhang[b]

[a] Key Laboratory of Low Dimensional Materials & Application Technology, Ministry of Education, and Faculty of Materials, Optoelectronics and Physics, Xiangtan University, Xiangtan 411105, PR China
[b] State Key Laboratory on Integrated Optoelectronics, and College of Electronic Science and Engineering, Jilin University, Changchun 130012, PR China

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ABSTRACT

$Bi_{0.5}Na_{0.5}TiO_3$–$Bi_{0.5}K_{0.5}TiO_3$ (BNT–BKT) powder is synthesized by a metal-organic decomposition method and characterized by field-emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD). A humidity sensor, which is consisted of five pairs of Ag–Pd interdigitated electrodes and an Al_2O_3 ceramic substrate, is fabricated by spin-coating the BNT–BKT powder on the substrate. Good humidity sensing properties such as high response value, short response and recovery times, and small hysteresis are observed in the sensing measurement. The impedance changes more than four orders of magnitude within the whole humidity range from 11% to 95% relative humidity (RH) at 100 Hz. The response time and recovery time are about 20 and 60 s, respectively. The maximum hysteresis is around 4% RH. The results indicate that BNT–BKT powder is of potential applications for fabricating high performance humidity sensors.

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1. Introduction

Chemical sensors are widely used in many fields such as environmental monitoring, inflammable-gas inspection, air-quality detection, and so on [1–3]. The investigation of high-performance chemical sensors is focused on the improvement of many sensor requirements, including linear response, high sensitivity, fast response speed, easy fabrication, and low cost [4–6]. Extensive researches have been done to develop novel sensing materials with various sensing properties [7–10]. Up to now, a series of materials, such as polymers, ceramics, and composites, have been proved to own their respective advantages and specific conditions for sensor application [11–13]. However, investigations on the synthesis and test of novel chemical sensing materials are still in great demand.

Humidity sensor, as an important field in chemical sensors, plays an important role in healthcare, agriculture, voyage and our daily life [13]. Recently, complex metal oxides such as $Na_2Ti_3O_7$ [14] and $LaCo_{0.3}Fe_{0.7}O_3$ [15] with special structures are found to own excellent humidity sensing properties. Simultaneity, their adjustable component and controllable microstructure make them very promising candidates for fabricating humidity sensors in practice.

Both $Bi_{0.5}Na_{0.5}TiO_3$ (BNT) and $Bi_{0.5}K_{0.5}TiO_3$ (BKT) are ABO_3-type complex metal oxides with the substitution of A-site ions, and the homogeneous solid solution of $Bi_{0.5}Na_{0.5}TiO_3$–$Bi_{0.5}K_{0.5}TiO_3$ (BNT–BKT) can be formed [16]. BNT–BKT has been extensively studied as piezoelectric materials, and also employed in lead-free piezoelectric micro-electro-mechanical system (MEMS) applications [17]. However, as far as our knowledge, relatively few reports on the humidity sensing properties of BNT–BKT powder are available in the literatures.

Previously, we have reported the humidity sensing properties of BKT powder [18]. This work aims at the improved sensing properties of BNT–BKT complex metal oxide. Compared with BKT, Na^+ ions in the BNT–BKT system make this material more sensitivity [19]. We expect the results offer useful guidelines to understand and employ the composite functional materials.

2. Experimental details

2.1. Preparation and characterization of BNT–BKT powder

The BNT–BKT precursor solution was prepared by first dissolving an appropriate amount of bismuth nitrate [$Bi(NO_3)_3 \cdot 5H_2O$] (99.5%), sodium nitrate [$NaNO_3$] (99%) and potassium nitrate [KNO_3] (99%) in acetic acid with a molar ratio of 2:1:1. Secondly, 2-methoxyethanol [$CH_3OCH_2CH_2OH$] and tetrabutyl titanate solution were added into the precursor solution. At last, acetylacetone [$CH_3COCH_2COCH_3$] (99%) was added into the mixture to prevent

* Corresponding author. Tel.: +86 731 58293648; fax: +86 731 58298119.
E-mail address: zhengxuejun@xtu.edu.cn (X. Zheng).

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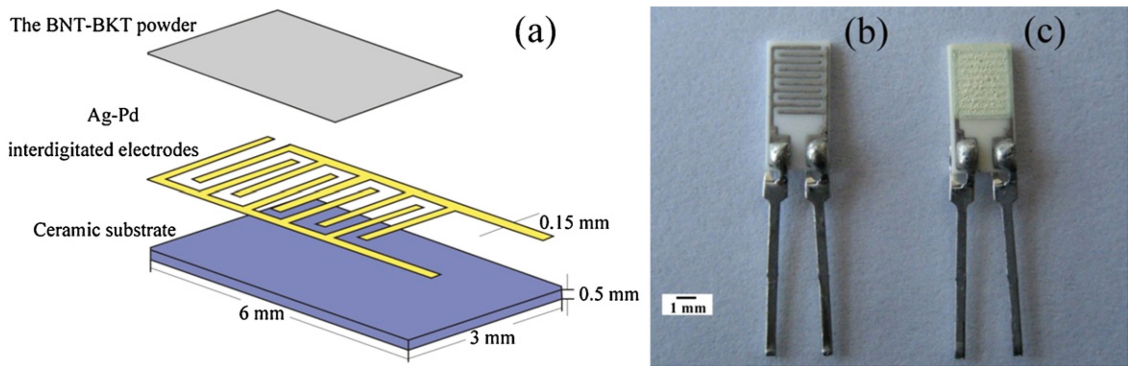

Fig. 1. (a) A schematic diagram of the humidity sensor structure, (b) top-view photograph of the blank sensor and (c) the sensor coated with the BNT–BKT powder.

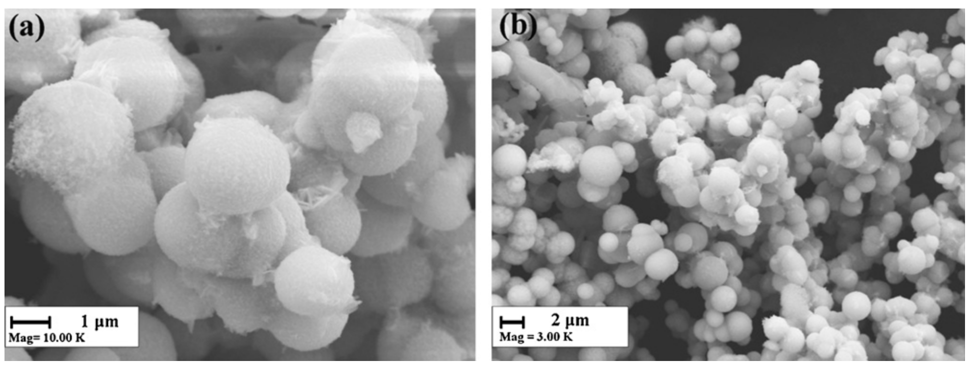

Fig. 2. (a) High-resolution FE-SEM image and (b) low-resolution FE-SEM image of the BNT–BKT powder.

the hydrolysis caused by the moisture in air. The obtained solution was stirred constantly until a transparent and stable light yellow precursor solution with a molar concentration of 0.25 mol/L was prepared. The precursor solution was annealed at 700 °C for 3 h. The resulting light yellow BNT–BKT powder was washed with deionized water and ethanol, and then dried at 60 °C in air for further characterization.

Field-emission scanning electron microscopy (FE-SEM) images were performed on a Carl Zeiss LEO1525 microscope with an accelerating voltage of 20 kV. Transmission electron microscopy (TEM) images were obtained on a JEOL JEM-2100 microscope operating at 200 kV. X-Ray diffraction (XRD) analysis was conducted on a Bruker-AXS model D8-ADVANCE X-ray diffractometer with Cu Kα radiation (λ = 1.5418 Å).

2.2. Fabrication and measurement of the humidity sensor

The main processes for fabricating BNT–BKT powder-based humidity sensors were as follows: firstly, the BNT–BKT powder was mixed with deionized water in a weight ratio of 100:25 to form a paste. Secondly, the paste was spin-coated onto an Al_2O_3 ceramic substrate (6 mm × 3 mm, 0.5 mm in thick) with five pairs of Ag–Pd interdigitated electrodes (electrodes width and distance: 0.15 mm) to form a sensing film (Fig. 1(a)), and then the film was dried in air at 60 °C for 5 h. Finally, the humidity sensor based on the BNT–BKT powder was obtained after aging at 95% relative humidity (RH) with a voltage of 1 V, 100 Hz for 24 h [20]. Fig. 1(b) and (c) show the photographs of the blank sensor and the sensor coated with the BNT–BKT powder, respectively.

A ZL-5 model LCR analyzer was used to measure the sensing characteristic curves of the BNT–BKT powder-based humidity sensors. The AC voltage applied was 1 V, and the frequency varied from 100 Hz to 100 kHz. To avoid the polarization effects of adsorbed water, the sensing properties of this ionic-type humidity sensor were evaluated by impedance changes [18]. The impedance value is a complex, which is composed of the resistance (real part) and the reactance (imaginary part). By using the ZL-5 model LCR analyzer, the complex impedance module and complex angle of the sensor impedance were obtained automatically, and the real part and imaginary part were calculated by using them.

The humidity generation sources were obtained through saturated salt solutions. The six different saturated salt solutions, including LiCl, $MgCl_2$, $Mg(NO_3)_2$, NaCl, KCl and KNO_3, were released in a closed glass vessel, and the corresponding RH values were 11%, 33%, 54%, 75%, 85% and 95% RH, respectively [21]. It takes 10 h for the air in the glass vessel to reach equilibrium state in the investigations. The RH of laboratory atmosphere was maintained at 25% RH by using an automatic drier. All of the sensing measurements were performed at a constant temperature of 25 °C. The response time and recovery time are defined as the time taken by a sensor to achieve 90% of the total impedance change in the case of adsorption and desorption [22].

3. Results and discussion

3.1. Morphology and structure analysis

The morphologies of the BNT–BKT powder are characterized by FE-SEM and shown in Fig. 2(a) and (b). It can be clearly seen that the BNT–BKT powder is of homogeneous spherical microstructure with diameters of 0.5–2 μm. Furthermore, as shown in Fig. 2(a), the surface of each spherical microstructure takes on a rough structure with some fragments attached on it. The appearance of the fragments is helpful for the increase of the specific surface, which is quite useful for the water molecule adsorption. The BNT–BKT powder is also featured by a high porosity as shown as Fig. 2(b), which is beneficial to their sensing properties [20].

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