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Humidity sensor based on electrospun $(Na_{0.5}Bi_{0.5})_{0.94}TiO_3$–$Ba_{0.06}TiO_3$ nanofibers

Peiwen Li[a,b], Xuejun Zheng[a,*], Yong Zhang[b,*], Mengjiao Yuan[b], Bin Jiang[b], Shuifeng Deng[b]

[a]School of Mechanical Engineering, Xiangtan University, Xiangtan 411105, PR China
[b]School of Physics and Optoelectronics, Xiangtan University, Xiangtan 411105, PR China

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Abstract

$(Na_{0.5}Bi_{0.5})_{0.94}TiO_3$–$Ba_{0.06}TiO_3$ (NBT–BT6) nanofibers were synthesized by electrospinning and characterized by X-ray diffraction, field emission scanning electron microscopy, and transmission electron microscopy. The humidity sensor was fabricated by coating NBT–BT6 nanofiber on a ceramic substrate with Ag–Pd interdigitated electrodes. At 100 Hz, the impedance of the humidity sensor changed by more than five orders of magnitude with the relative humidity (RH) changing from 11% to 95%. The response time and recovery time were about 2 s and 50 s, respectively, and the maximum hysteresis was around 5% RH. The results indicate that NBT–BT6 nanofibers are of great potential for high-performance humidity sensor applications.
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Keywords: $(Na_{0.5}Bi_{0.5})_{0.94}TiO_3$–$Ba_{0.06}TiO_3$ nanofibers; Electrospinning; Humidity sensor; Complex impedance plots

1. Introduction

Humidity sensors have attracted much attention for their diverse applications in industrial, agricultural, aeronautics, medicine and meteorology [1–5]. In recent years, more and more researchers are paying their attention to exploitation of materials for humidity sensor with excellent humidity sensing properties including high sensitivity, linear response, and fast response speed [6–9]. One-dimensional (1D) nanostructures, such as nanofibers, nanowires, and nanotubes, have been gained considerable attention in humidity sensing because of their special physical and chemical properties [10–14]. Electrospinning is a relatively simple and low-cost technique for synthesis of nanofibers [15,16], and the nanofibers synthesized by electrospinning have been identified as good candidates for fabricating high-performance humidity sensors [17–19].

Perovskite-type ABO_3 oxides, in which A are rare earth, alkali and alkaline earth metal ions and B are transition metal ions [8,20,21], have been recognized as a promising humidity sensing material due to their good chemical and thermal stability [4,22,23]. Substitution of proper metal ions in perovskite-type ABO_3 oxides can improve the humidity sensing properties [6,24]. The $(Na_{0.5}Bi_{0.5})_{(1-x)}TiO_3$–$Ba_xTiO_3$ ($(1-x)$NBT–BTx), as an ABO_3-type complex metal oxide with the substitution of A-site ions, has been studied by many researchers. Its electrical properties can be affected by the compositions of $(1-x)$NBT–BTx [25]. According to previous work [26,27], there is low dielectric loss for the $(1-x)$NBT–BTx at a composition around x=0.06, and the materials with low dielectric loss and high permittivity would be used for fabricating the humidity sensors with good insulating properties and reliability [28]. In order to improve the humidity sensing properties, NBT–BT6 nanofibers were synthesized by the electrospinning technique which would increase its specific surface area.

In this paper, the $(Na_{0.5}Bi_{0.5})_{0.94}TiO_3$–$Ba_{0.06}TiO_3$ (NBT–BT6) nanofibers were synthesized by electrospinning, and the humidity sensor based on NBT–BT6 nanofiber was fabricated for the investigation of humidity sensing properties. The crystalline structure and morphology of NBT–BT6 nanofibers

*Corresponding authors. Tel./fax: +86 731 58293210.
E-mail addresses: zhengxuejun@xtu.edu.cn (X. Zheng), zhangyong@xtu.edu.cn (Y. Zhang).

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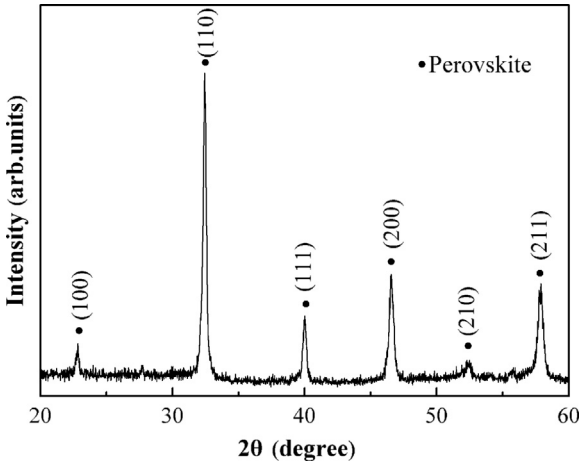

Fig. 1. XRD pattern of NBT–BT6 nanofibers.

were characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and transmission electron microscopy (TEM). The humidity sensing properties were obtained via impedance measurement of NBT–BT6 nanofiber humidity sensor at different relative humidity (RH) and various frequencies. We expect the NBT–BT6 nanofiber would be hopeful candidate for fabricating high-performance humidity sensors.

2. Experimental

2.1. Synthesis and characterization of NBT–BT6 nanofibers

The preparation of NBT–BT6 precursor solution is reported in our previous work [15]. The PVP solution was prepared by adding 1.4 g of polyvinyl pyrrolidone (PVP) (MW = 1,300,000) in 10 mL ethanol under vigorous stirring for 6 h. The NBT–BT6 precursor solution and the PVP solution were mixed and vigorously stirred for 12 h to form a homogeneous solution. The resulting solution was loaded into a plastic syringe with a needle, and then electrospinning was performed using a computer-controlled syringe pump. Composite (NBT–BT6/PVP) nanofibers were collected on an aluminum foil with an applied voltage of 20 kV. The distance between syringe needle tip and collector was 17 cm. The as-spun nanofibers were calcined in air at 700 °C for 1 h. The crystalline structure of NBT–BT6 nanofibers was characterized by XRD (Brukers, D8 Advance) with Cu Kα ($\lambda = 0.15418$ nm) radiation. The morphology was characterized by FE-SEM (Hitachi, S-4800) and TEM (JEOL, JEM-2100).

2.2. Fabrication and measurement of the humidity sensor

The NBT–BT6 nanofibers were mixed with deionized water to form a paste and it was coated on a ceramic substrate (6.8 mm × 3 mm, 0.66 mm in thick) with 5 pairs of Ag–Pd interdigitated electrodes (electrode width and distance: 200 μm) to form a sensing film, and then the sensing film was dried in air. The film thickness was about 150 μm, which was measured by an AMBIOS XP-2 step profiler. According to the previous results [29–31], the film thickness can affect the sensor response and dynamics. The humidity sensing properties of NBT–BT6 nanofiber humidity sensor were measured on an intelligent humidity sensing analysis system (Beijing Elite Tech Co., Ltd, CHS-1). The applied voltage was AC 1 V, and the range of 11–95% RH was obtained using saturated salt solutions as the humidity generation sources [32]. The six different saturated salt solutions were LiCl, $MgCl_2$, $Mg(NO_3)_2$, NaCl, KCl, and KNO_3, and their corresponding RH values were 11%, 33%, 54%, 75%, 85% and 95% RH, respectively. The NBT–BT6 nanofiber humidity sensor was put into six glass chambers with different RH levels at room temperature to investigate the humidity sensing properties, such as sensitivity characteristic, the response and recovery behaviors, and hysteresis characteristic.

3. Results and discussion

3.1. Structural analysis and morphology characterization

The XRD pattern of NBT–BT6 nanofibers is shown in Fig. 1. The XRD pattern exhibits a single-phase perovskite structure without a second phase, and the diffraction peaks were indexed according to standard diffraction pattern data of the NBT phase compiled in JCPDS card (JCPDS, 46-0001). Furthermore, the average crystallite size was calculated from X-ray line broadening analysis based on the Scherrer equation $D = 0.89\lambda/(\beta\cos\theta)$, where D is the average crystal size in nm, λ is the X-ray wave length ($\lambda = 0.1541$ nm), β is the full width at half-maximum of the peak, and θ is the corresponding Bragg diffraction angle [33]. The average crystalline size of NBT–BT6 nanofibers is calculated as 29.98 nm. The diffraction peaks are sharp and narrow, indicating a good crystalline phase of NBT–BT6 nanofibers.

The low magnification FE-SEM image of NBT–BT6 nanofibers is shown in Fig. 2(a). The NBT–BT6 nanofibers are interlaced with each other to form network structure, and the network structure is helpful for the adsorption of water molecules, which is beneficial to humidity sensing properties [34]. The high magnification FE-SEM image is shown in inset of Fig. 2(a). The NBT–BT6 nanofiber is of uneven diameter and it is about 110 nm. In order to further investigate the morphology of NBT–BT6 nanofibers, the TEM image is shown in Fig. 2(b). It can be seen that the surfaces of nanofiber are rough because the nanofibers are composed of crystal grains with different size. The tips of the nanofibers and necks between crystal grains would affect the accumulation of charge that present a high local charge density to promote water dissociation, which are benefit to humidity sensing properties. [4,34].

3.2. Humidity sensing properties of NBT–BT6 nanofibers

Fig. 3 shows the dependence of impedance on the RH for NBT–BT6 nanofiber humidity sensor at different frequencies. At low RH, the impedance decreases remarkably with the increasing frequency, while it decreases slowly with the increasing frequency

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