



# Mg<sup>2+</sup>/Na<sup>+</sup>-doped rutile TiO<sub>2</sub> nanofiber mats for high-speed and anti-fogged humidity sensors

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## ABSTRACT

Mg<sup>2+</sup> and Na<sup>+</sup> doped rutile TiO<sub>2</sub> nanofibers have been prepared through in situ electrospinning technique and calcination with poly(vinyl pyrrolidone) (PVP) nanofibers as sacrificed template. The as-prepared composite nanofibers are spin-coated onto a ceramic substrate with three pairs of carbon interdigital electrodes to measure its humidity sensing behaviors. The product exhibits high-speed response (2 s) and recovery (1 s) for detecting moisture. Additionally, under UV irradiation, a water contact angle ( $\theta$ ) of nearly 0° has been observed based on the product, providing our humidity sensor with the anti-fogged properties.

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## 1. Introduction

The past several decades have witnessed the huge progress on the fabrication of humidity sensors for their practical applications in air-quality control, environmental monitoring, healthcare, defense and security, etc. [1,2]. Recently, taking the advantages of large surface area, high surface to volume ratio, and special physical and chemical properties of one-dimensional (1D) metallic oxide nanostructures, the synthesis of sensitive humidity sensors based on 1D metallic oxide nanostructures is of current interest [3–7]. Hitherto, many sensitive and stable humidity sensors have been reported. For example, Fu and Wang reported the fast humidity sensors based on pure CeO<sub>2</sub> nanowires [3]. Zhang and Wang studied the humidity sensing performances based on Ba-doped CeO<sub>2</sub> nanowires [4]. Mathur and coworkers used the individual tin oxide nanowires to detect the water vapor in different gaseous environments [5]. Wang and co-workers investigated the humidity sensing properties based on a single SnO<sub>2</sub> nanowire [6]. Our group presented the highly sensitive and stable humidity nanosensors based on LiCl doped TiO<sub>2</sub> electrospun nanofibers [7]. Although huge and significant progresses on humidity sensors have been obtained,

high-speed humidity sensor with both response time and recovery time less than 2 s, during the whole relative humidity measurement, has still not been obtained yet. Additionally, no papers on anti-fogged and high-speed humidity sensors have been reported.

TiO<sub>2</sub>, as an important metallic oxide, has been widely investigated in the fields of environmental cleaning [8] and protection [9], sensors [10], anti-fogged mirror [11], and solar cells [12]. Recently, rutile TiO<sub>2</sub> (1 1 0) has also been proven to be active sites for water dissociation [13]. Those outstanding properties make rutile TiO<sub>2</sub> a good candidate in synthesizing humidity sensors. Mg<sup>2+</sup> and Na<sup>+</sup> are often used in humidity sensors to facilitate the ionic conduction during the humidity sensing measurement [14,15]. In this paper, 1D rutile TiO<sub>2</sub> nanofiber, Mg<sup>2+</sup>, and Na<sup>+</sup> are combined, for the first time, to form a novel type high-speed and anti-fogged humidity sensor via electrospinning and calcination. The response time and recovery time, during the whole relative humidity (RH: 11–95%), are 2 s and 1 s, respectively. Most importantly, under UV irradiation, a water contact angle ( $\theta$ ) of nearly 0° has been observed based on the product, endowing our humidity sensor with the anti-fogged properties.

## 2. Experimental

### 2.1. Chemical

MgCl<sub>2</sub> (>95%), tetrabutyl titanate (>95%), ethanol (>95%) and acetic acid (>95%) were of analytical grade and purchased

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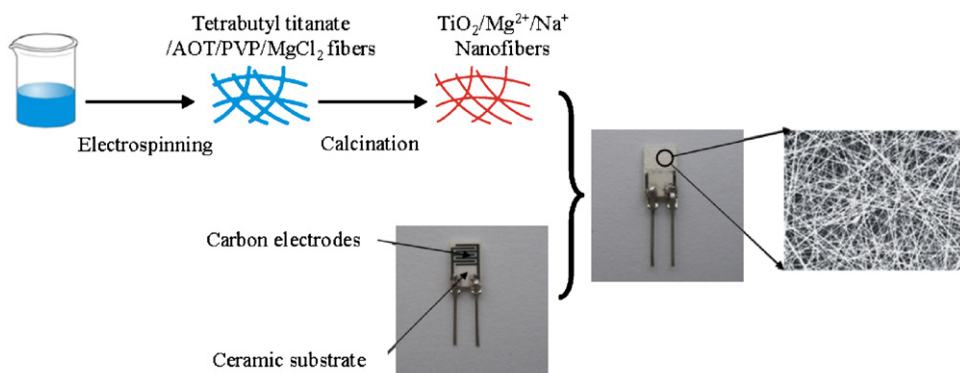


Fig. 1. Schematic diagrams of the steps to fabricate  $\text{Mg}^{2+}/\text{Na}^{+}$ -doped  $\text{TiO}_2$  nanofiber mats for humidity measurement via electrospinning and calcination.

from Tianjin Chemical Company. Poly(vinyl pyrrolidone) (Mw: 1,300,000) and dioctyl sulfosuccinate sodium (AOT:  $\text{C}_{20}\text{H}_{37}\text{OSNa}$ ) were purchased from Aldrich.

## 2.2. The whole procedures for humidity sensor

In a typical procedure, 1.5 g of tetrabutyl titanate was mixed with 3 mL of acetic and 3 mL of ethanol in glovebox under vigorous stirring for 10 min. Subsequently, this solution was added to 7.5 mL of ethanol containing 0.45 g of poly(vinyl pyrrolidone) (PVP), 0.02 g of dioctyl sulfosuccinate sodium (AOT:  $\text{C}_{20}\text{H}_{37}\text{OSNa}$ ) and a suitable amount of  $\text{MgCl}_2$  under vigorous stirring for 30 min. The mixture was loaded into a glass syringe and connected to high-voltage power supply for electrospinning, 12 kV was provided between the cathode (a flat aluminum foil) and anode (syringe) at a distance of 20 cm. Then, calcination ( $600^\circ\text{C}$  in air for 3 h) has been used to remove PVP and to convert both tetrabutyl titanate into rutile  $\text{TiO}_2$  nanofibers and AOT into  $\text{Na}^{+}$  according to previous reported method [16].

Consequently, the as-prepared  $\text{Mg}^{2+}/\text{Na}^{+}$  doped  $\text{TiO}_2$  nanofibers were mixed in a weight ratio of 100:5 and were ground with deionized water to form a dilute paste. The paste was screen-printed onto a ceramic substrate ( $7\text{ mm} \times 5\text{ mm}$ , 0.5 mm in thick) with three pairs of carbon interdigital electrodes (electrodes width and distance: 0.15 mm) to form a film with the thickness about  $10\ \mu\text{m}$ , and then the film was dried at  $60^\circ\text{C}$  in air for 5 h. Finally, the humidity sensor was fabricated after aging at 95% RH with a voltage of 1 V, 100 Hz for 24 h. The characteristic curves of humidity sensitivity were measured on a ZL5 intelligent LCR test meter (Made in Shanghai, China) at room temperature. The voltage applied in our studies was AC 1 V. The controlled humidity environments were achieved using supersaturating aqueous solutions of different salts of LiCl,  $\text{MgCl}_2$ ,  $\text{Mg}(\text{NO}_3)_2$ , NaCl, KCl and  $\text{KNO}_3$  in a closed glass vessel at room temperature, which yielded 11%, 33%, 54%, 75%, 85% and 95% RH, respectively. This method was established by Wang [17,18]. The procedures for the preparation of the new sensors are illustrated in Fig. 1.

## 2.3. Measurements

The X-ray powder diffraction (XRD) data were collected on an X'Pert MPD Philips diffractometer ( $\text{Cu K}\alpha$  X-radiation at 40 kV and 50 mA). Scanning electron microscopy (SEM) images were recorded on a SHIMADZU SSX-550 (Japan) instrument. The humidity measured machine was ZL5 intelligent LCR test meter made in Shanghai China. Water CA was measured with an OCA20 (Dataphysics, Germany) at ambient temperature.

## 3. Results and discussion

### 3.1. Materials characterization

Fig. 2a shows the SEM image of the  $\text{TiO}_2$  nanofibers containing 5.56 wt.%  $\text{Mg}^{2+}$  and 0.22 wt.%  $\text{Na}^{+}$ , indicating a large scale of composite nanofibers with the average diameter of 200 nm can be obtained via our method. In XRD pattern, as shown in Fig. 2b, (1 1 0), (1 0 1), (2 0 0), (1 1 1), (2 1 0), (2 1 1), (2 2 0), (0 0 2), (3 1 0), (3 0 1) and (1 1 2) peaks at  $2\theta = 27.35^\circ$ ,  $36.0^\circ$ ,  $39.15^\circ$ ,  $41.2^\circ$ ,  $44^\circ$ ,  $54.25^\circ$ ,  $56.55^\circ$ ,  $62.75^\circ$ ,  $63.9^\circ$ ,  $68.95^\circ$ , and  $69.8^\circ$  can be clearly detected correspond-

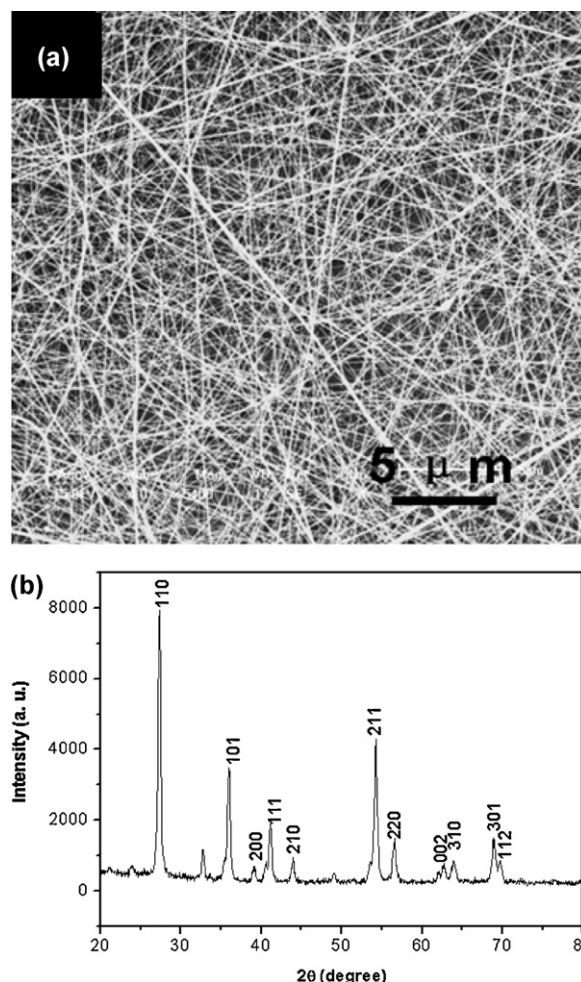


Fig. 2. (a) SEM image and (b) XRD pattern of the as-prepared product.

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