## The preparation of C-N co-doping Zirconia electrospun nanofibers and their humidity sensing properties

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The chemical states of constituent elements in C-N/ZrO<sub>2</sub> composites were characterized by X-ray photoelectron spectroscopy (XPS). Taking C-N/ZrO<sub>2</sub>-3 as the example to illustrate, as shown in Fig. S1, the presence of Zr, O, C, and N in C-N/ZrO<sub>2</sub>-3 composite are confirmed in the XPS survey spectrum (Fig. S1a). The coexistence of Zr 3d<sup>3/2</sup> and Zr 3d<sup>5/2</sup> in the nanocomposites is evidenced by the peaks at 182.3 eV and 184.6 eV (Fig. S1b), which is in good agreement with that the reported values for Zr<sup>4+</sup> states in ZrO<sub>2</sub> species.<sup>1-3</sup> The C 1s and N 1s spectra in the C-N/ZrO<sub>2</sub>-3 are displayed in Fig. S1 (c) and Fig. S1 (d), respectively. The three peaks of C 1s are located at 284.9 eV, 286.4 eV and 288.7 eV. The peak at 286.4 eV and 288.7 eV can be identified as the signal of sp<sup>2</sup> carbon C-C or C-N.<sup>4</sup> The peaks at 286.4 eV and 288.7 eV can be ascribed to C-O and C-N-C species.<sup>5</sup> The N 1s spectrum exhibits three obvious peaks at 398.4 eV, 400.1 eV and 404.1 eV, which are assigned to C=N-C, pyrrolic nitrogen, and uncondensed amino functional groups respectively.<sup>5, 6</sup> The obtained characterization results by XPS are consistent with the FT-IR, which means C and N

have been successfully introduced into zirconia.



Fig. S1 XPS survey spectrum (a) and the core level spectra of (b) Zr 3d (c) C 1s and (d) N 1s of C-  $N/ZrO_2\mathchar`-3$ 

As we all know, it is essential to explore the optimum working frequency of the humidity test for materials. Fig. S2 provides the impedances of  $ZrO_2$  and C-N/ $ZrO_2$ -x (x=1, 2, 3, 4, 5) composites which have been investigated at different frequencies of 10 Hz-100 kHz. At low frequency, there is a lack of data at 11% RH in the humidity sensing curves, due to its extremely high resistance, which has overpassed the detection limit for every material. At high frequency, the order of magnitude change of the curves is not obvious. By comparison, it is discovered that the humidity sensing curve at 100 Hz shows the best linearity and the largest change of magnitude among all the working frequencies. Therefore, 100 Hz was selected as the operating frequency in the subsequent humidity sensing tests.



Fig. S2 The impedance of sensor based on (a)  $ZrO_2$ , (b) C-N/ $ZrO_2$ -1, (c) C-N/ $ZrO_2$ -2, (d) C-N/ $ZrO_2$ -3, (e) C-N/ $ZrO_2$ -4 and (f) C-N/ $ZrO_2$ -5 varied with a series of humidity at frequencies from 10 Hz to 100 kHz.

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