An Easy Method of Preparing Ozone Gas Sensors based on ZnO

Nanorods

1 – Preparation: Hydrothermal treatment method

Growth of the 1-D ZnO nanorods was accomplished using the a hydrothermal treatment method. A detailed description of the hierarchical 1-D ZnO nanorod-like structure growth onto SiO$_2$/Si substrate is presented below. All reagents were obtained from the Aldrich company, exhibiting > 98%.

To seed layer was deposited from a polymeric ZnO resin, which was synthesized via polymeric precursor method. Zinc nitrate (Zn(NO$_3$)$_2$.6H$_2$O) was dissolved in deionized water and mixed with an aqueous citric acid (CA) solution under constant stirring at 100 °C, while. The molar ratio of total metal ions to CA was maintained at 1:3. The temperature was increased to 150 °C and ethylene glycol (EG) was added with an EG:CA mass ratio of 60:40. The resin viscosity was adjusted by water evaporation until it reached 12 cP (centipoise) using a rheometer (Brookfield, LVDV-III ULTRA). Then the solution was spin-coated onto a SiO$_2$/Si (with Pt electrodes) substrate at a speed of 10000 rpm for 60 s, followed by water evaporation at 100°C for 30 min. A two-stage heat treatment was performed in which the sample was: initial heated at 300°C for 30 min with a heating rate of 1°C.min$^{-1}$ in air atmosphere to pyrolyze the organic compounds, then heat-treatment for 2 hours at 500°C with a heating rate of 5°C.min$^{-1}$ for crystallization process.

To obtain the 1-D ZnO nanorod-like structures, a solution was prepared by using zinc nitrate and hexamethylenetetramine, which were dissolved into a de-ionized water
under vigorous stirring at room temperature and placed in screw-vapped bottle. Then the substrate containing the ZnO seed layer was immersed in the solution with the ZnO seed layers face down (Fig.1S(a)). ZnO nanorods were hydrothermally grown by maintaining the as screw-capped bottle in a furnace for 4 hours at 110°C with a heating rate of 10°C.min⁻¹. At the end of the hydrothermal treatment, the sample was removed from the solution (Fig.1S(b)), thoroughly washed several times with deionized water and isopropyl alcohol to remove white loosely adherent powder precipitated during deposition, and dried overnight at 80°C.

Figure 1S. Photograph of glass bottle used to grow ZnO nanorod-like structures and the SiO₂/Si substrates containing the ZnO seed layer. (a) Before and (b) after hydrothermal treatment for 4 hours at 110°C.
2 – X-ray absorption near edge spectroscopy (XANES) measurements

X-ray absorption near edge spectroscopy (XAS) data was collected at the Brazilian Synchrotron Light Laboratory (LNLS), using XAFS2 beamline. This characterization technique was applied in order to probe the local order around Zn atoms in hierarchical 1-D ZnO nanorod-like structures. The Zn K-edge XANES spectrum was collected in the fluorescence mode, at room temperature, using a 15-element Ge solid-state detector (Ge-15). This spectrum was recorded between 9600 and 9750 eV using energy step of 0.3 eV around the edge. The XANES spectrum was processed using the Multiplatform Applications for XAFS code (MAX).1

![Figure 2S. Zn K-edge XANES spectrum of ZnO nanorods.](image)
3 – Energy dispersive X-ray (EDX) spectroscopy analysis

Figure S3 shows the EDX spectrum of the ZnO nanorod-like structures prepared via hydrothermal treatment at 110°C.

Figure 3S- Energy dispersive X-ray (EDX) spectroscopy analysis of the ZnO nanorods.
4 – Electrical resistance measurement

Figure 4S shows the variation in the electrical resistance of ZnO nanorods after switching on the UV illumination. After UV-light exposition, the electrical response of ZnO nanorod-like structures is quickly decreased.

**Figure 4S.** Electrical response of ZnO nanorods under UV-light illumination.

REFERENCES