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Nanostructures of tungsten trioxide, nickel oxide and niobium oxide for chemical sensing applications

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**Abstract**

Tungsten trioxide nanowires, nickel oxide nanowires and niobium oxide nanostructures were synthesized by thermal oxidation, vapour-liquid-solid (VLS) technique and hydrothermal method, respectively. Alumina ( $2 \cdot 2 \text{ mm}^2$ ) substrates were used for growth of the different metal oxides nanostructures. Thin films of tungsten or niobium are used as source material for  $\text{WO}_3$  and  $\text{Nb}_2\text{O}_5$ . An ultrathin film of gold was deposited on alumina as catalyst for NiO. All these materials were deposited by RF magnetron sputtering. The morphology was investigated by scanning electron microscope and the functional properties of these structures were tested towards several gaseous species at different working temperatures. The results obtained show that these sensing materials have similar performances considering the different n- and p-type semiconductors behaviour. Tungsten trioxide ( $\text{WO}_3$ ) nanowires, nickel oxide (NiO) nanowires and niobium oxide ( $\text{Nb}_2\text{O}_5$ ) nanostructures were synthesized by thermal oxidation, vapor-liquid-solid (VLS) technique and hydrothermal method, respectively. The morphology was investigated by scanning electron microscope and the functional properties of these structures as gas sensors were tested for several gaseous species at different working temperatures. In this work a comparison between the sensitivity of these devices towards acetone is shown.

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## 1. Introduction

Nowadays metal oxide nanostructures are widely investigated in the field of gas sensing thanks to their high ability to recognize different gaseous species. The growth of these nanostructures can be made by applying different methods to the bulk initial material, that is in most cases a metal foil or powder. Prepared nanostructures must be transferred on an appropriate transducer in order to be used as gas sensors.

One-dimensional structures, like nanowires, are characterized by high crystallinity that allows a reduction of the problem of stability. For this reason, gas sensors based on metal oxide one-dimensional nanostructures are integrated in several sensing devices such as electronic nose [1, 2].

This work shows the behaviour of three different types of gas sensors based on metal oxide nanostructures. They were obtained using three synthesis methodologies, in particular thermal oxidation, hydrothermal treatment and VLS technique for  $\text{WO}_3$ ,  $\text{Nb}_2\text{O}_5$  NiO respectively. The growth was obtained directly on the active transducer, without needing to transfer the nanostructures after the synthesis process. The gas sensing properties of these devices were tested toward different target gases (ethanol, acetone, CO and  $\text{NO}_2$ ), and this work focuses on sensors responses to acetone, making a comparison between their performances in terms of sensitivity as a function of gas concentration and operating temperature.

## 2. Experimental

Alumina ( $2 \cdot 2 \text{ mm}^2$ ) substrates were used for growth of all three different metal oxide materials. At first, they were cleaned for 15 minutes in acetone, to remove the deposited particles or any organic compound, and then dried using synthetic air. Afterwards, three different growth mechanisms were used for the synthesis of the nanostructures of each metal oxide.

### 2.1. $\text{WO}_3$ nanowires growth by thermal oxidation

The growth process starts by depositing a layer of metallic tungsten (the thickness 180 nm) on the alumina substrates using RF magnetron sputtering. Afterwards, the samples were oxidized in a tubular furnace at  $550 \text{ }^\circ\text{C}$ , with a pressure of 0.8 mbar, with 2 sccm oxygen flow to promote the growth of the  $\text{WO}_3$  nanowires [3].

### 2.2. NiO nanowires growth by VLS technique

At first, an ultrathin layer of gold catalyst was deposited on the alumina substrates using RF magnetron sputtering. Then samples were placed into a tubular furnace for the growth of NiO nanowires. The NiO powder was heated to temperature of  $1400 \text{ }^\circ\text{C}$  to make it evaporate, to form vapours at pressure of 1 mbar, while the substrates were placed at temperature of  $930 \text{ }^\circ\text{C}$ . Argon gas (100 sccm) was used as carrier gas to transport the vapours of NiO towards the substrate, so they condensate in the form of nanowires on the substrates.

### 2.3. $\text{Nb}_2\text{O}_5$ nanostructures growth by hydrothermal method

A thin layer of niobium (500 nm) was deposited on the alumina substrates using RF magnetron sputtering. As prepared samples were placed in an autoclave with 0.8 mL of 0.01 M KOH solution for 10 hours at temperature of  $175 \text{ }^\circ\text{C}$ . After this treatment samples surface was covered with a white compound. The samples were treated with  $\text{HNO}_3$  and annealed at  $650 \text{ }^\circ\text{C}$  under argon flow to obtain the  $\text{Nb}_2\text{O}_5$  nanostructures [4].

### 2.4. Morphological characterization

After the growth process, samples surface was investigated by a field effect emission scanning electron microscope (LEO 1525) operated at 3-7 kV beam voltage.

2.5. Gas sensing

Resistance-conductance variation due to the exposure of different gases was measured as the response of each sensor. In prior to fabrication of the sensing devices, a platinum heating element on the back of every sample and platinum contacts on top of the nanostructures were deposited by DC magnetron sputtering. Gold wires were used to connect the samples on TO package. Then, the devices were placed in the test chamber to investigate the electrical response towards target gas at different concentrations and at various working temperatures. The measurements were obtained by keeping the chamber at 20 °C, with relative humidity of 50% and applying 1 V to the electrode of each sensor devices.

3. Results

The three growth processes applied to the samples gives different nanostructures, which are investigated by SEM. Fig. 1 shows the morphology of WO<sub>3</sub> nanowires (a), NiO nanowires (b) and Nb<sub>2</sub>O<sub>5</sub> nanostructures (c). From this investigation tungsten trioxide and nickel oxide nanowires were found thin and dense on the other hand niobium oxide nanostructures have flower like structures covers the surface uniformly.

The performances of the each sensor device were tested towards acetone in 10-100 ppm concentration range. Fig. 2 (a) shows the response of all the sensors to 100 ppm of acetone at wide range of temperatures. The graph underlines that the sensors have different optimal working temperatures, in particular WO<sub>3</sub> shows the best response at 400 °C, NiO at 500 °C and Nb<sub>2</sub>O<sub>5</sub> at 550 °C.

Fig. 2 (b) gives information about the trend line of the three different sensors at different concentrations of acetone (10, 30, 100 ppm) at the optimal working temperature for each device. However, the optimal operative temperature of the sensors is different, but the slops of the calibration curves are very similar. The data from these calibration curves, we had calculated the detection limits for each sensor considering minimum response as 1. The detection limits were found out be 0.07 ppm, 0.32 ppm and 4.88 ppm for WO<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub> and NiO respectively.

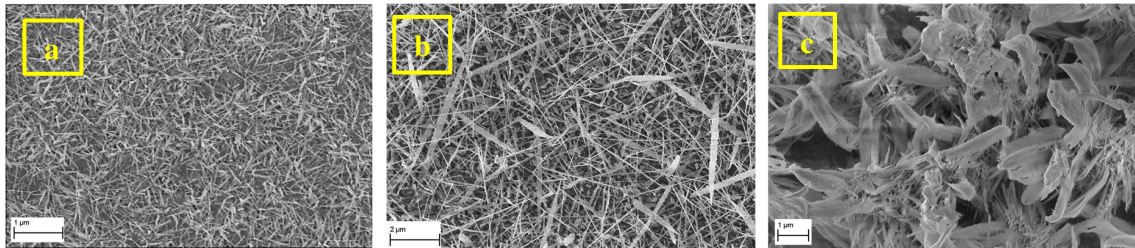


Fig. 1. Surface morphology of WO<sub>3</sub> nanowires (a); NiO nanowires (b); Nb<sub>2</sub>O<sub>5</sub> nanostructures (c).

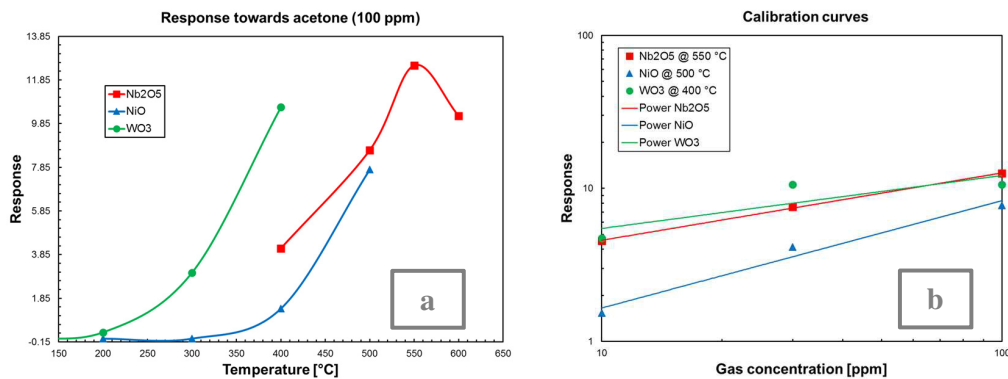


Fig. 2. (a) Response towards acetone 100 ppm; (b) calibration curves

#### 4. Conclusions

The detection of acetone is important in health care such as for diabetes. In fact, people suffering from this pathology have a particular breath odor, due to the production of a large amount of acetone through their normal metabolic processes.

In this work are shown three different chemical sensors that are able to recognize different concentrations of acetone. Thanks to this ability, they might find out a natural application in sensing devices to cure diseases.

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