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Nickel oxide nanowires growth by VLS technique for gas sensing application

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Abstract

In this work, nickel oxide (NiO) nanowires have been synthesized on alumina substrate by vapor-liquid-solid (VLS) technique and were tested for alcohol sensing. RF magnetron sputtered gold particles were used as catalyst for the growth of NiO nanowires. Nanowire surface morphology was analyzed by using scanning electron microscopy (FE-SEM). Afterwards, a set of conductrometric sensing devices were prepared and mounted on TO packages using electro-soldered gold wires. The response of prepared sensors towards different gases such as acetone and ethanol was measured. The dynamic sensing phenomena and the calibration curves of NiO nanowire sensors exposed towards these gases were also studied in detail. The optimal working temperature of the sensors were found to be 500°C for both the gases.

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1. Introduction

Recently, one dimensional (1D) metal oxide based nanostructures have attracted great deal of attention in the field of gas sensors due to their remarkable physical and chemical properties, significantly different from bulk materials. Furthermore, gas sensors based on 1-D nanostructures of n-type metal oxide semiconductors have been widely investigated in literature [1]. On the contrary, p-type metal oxide semiconductors have not yet been extensively studied for gas sensing applications. Nickel oxide (NiO) has cubic rock salt structure and is considered as a p-type semiconductor with wide band gap [2]. A variety of different techniques such as template assisted method, sol-gel method etc. were used to synthesize NiO nanowires. However, to best of our knowledge, there have been no reports available in literature on the growth of nickel oxide nanowires using VLS technique for the gas sensing applications. Here we are presenting a synthesis of NiO nanowires using VLS technique for gas sensing applications.

2. Experimental

2.1. Preparation of substrates

Alumina substrates (2mm×2mm) were used to grow the NiO nanowires. The substrates were cleaned in ultrasonic cleaner with acetone for 15 min and dried with synthetic air. The deposition of an ultrathin layer of catalyst on alumina substrates was performed by using magnetron sputtering (Kenotec Sputtering system). Herein we had used noble metal gold as a catalyst. Different deposition parameters such as 50W argon plasma, 5×10^{-3} mbar, 7 SCCM Ar flow were used for deposition of gold catalyst. These catalyst particles play an important role in promoting the nucleation sites during growth process.

2.2. VLS process for growth of NiO nanowires

Nickel oxide nanowires were directly grown on catalyst deposited substrate using VLS technique. The basic mechanism of VLS technique consists the formation of liquid alloy of metal catalyst by adsorbing vapors of source material at certain temperature. As these vapors are continuously provided, liquid alloy starts to saturate and these solid precipitate with time grows as 1D nanostructure [3]. To carry out this process an alumina tubular furnace (Custom design based on a Lenton furnace) shown in the Fig. 1 was used. NiO powder was used as source material. The powder was heated at 1400°C to induce evaporation and pressure inside the alumina tube was kept at 1 mbar. The substrates were placed in comparable low temperature region at 930°C. 100 SCCM of argon gas was used to transport vapors of NiO towards catalyst deposited alumina substrates. As the evaporated material reaches low temperature region, it starts condensates on the substrates in the form of NiO nanowires.

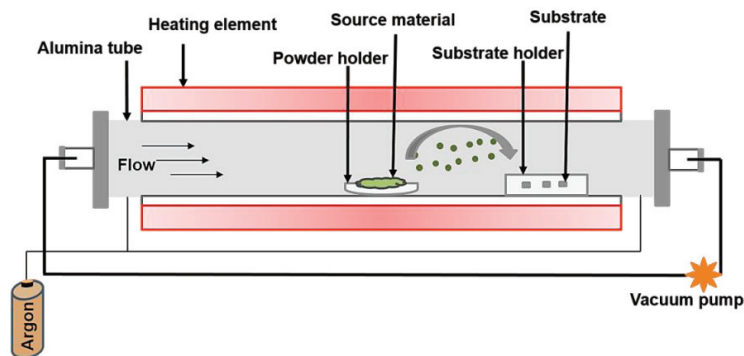


Fig. 1. The Schematic diagram of growth setup (Tubular Furnace).

2.3. Scanning electron microscopy

The surface morphology of deposited NiO nanowires was examined using SEM (Zeiss, Leo1525 Gemini model; Carl Zeiss AG, Oberkochen, Germany) operated at 5kV. Samples were fixed on metallic stub with a carbon-based glue to reduce sample charging during observation.

2.4. Device fabrication and Gas sensing

For sensor device fabrication a set of identical samples of NiO nanowires were prepared on alumina substrate. The interdigitated platinum contact was deposited using DC magnetron sputtering. On the other side of the alumina substrates a heating element was deposited to study the behavior of sensors at different working temperatures. The prepared devices were finally mounted on TO packages using electro-soldered gold wires. To investigate the conductometric response of these sensors in the presence of different gases species, a home-made test chamber was used. Different types and concentrations of gases were used, and the response of the sensors were recorded in a wide range of temperature (200°C-500°C). The total flow inside chamber was 200SCCM mixed with the synthetic air. Measurements were obtained by keeping the climatic chamber at 20°C, with a relative humidity equal to 50% and applying 1V to the sensor devices.

3. Results

Fig 2 shows the morphology of NiO nanowires at different magnification. The nanowires exhibit dense morphology with a high length-diameter ratio, with diameter in range of 16nm to 50nm.

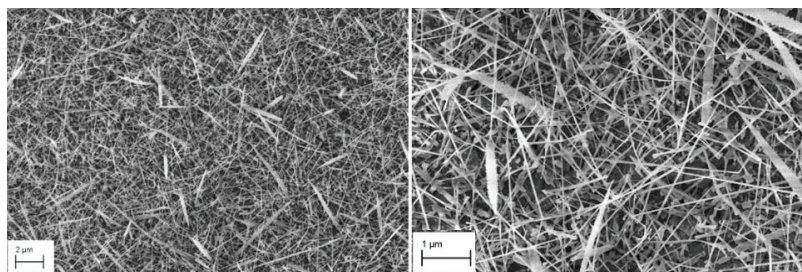


Fig. 2. SEM image of nickel oxide nanowires (NiO) prepared by VLS technique.

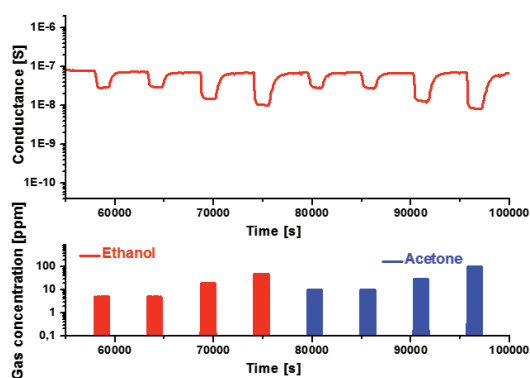


Fig. 3. Dynamic response of NiO Nanowire sensor towards reducing gases, ethanol red color; 5-5-20-50 ppm at 500°C and acetone blue color; 10-10-30-100 ppm at 500°C. Relative humidity of 50% @ 20°C.

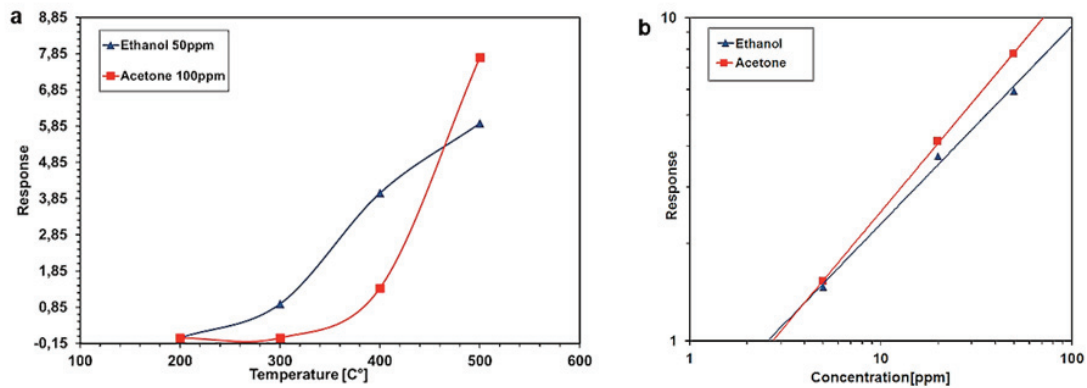


Fig. 4. (a) Response of NiO nanowires sensors towards Acetone (red curve) 100ppm and Ethanol (blue curve) 50ppm; (b) Calibration curves drawn towards Acetone (red curve) and Ethanol (blue curve) at optimal working temperature of 500°C. Relative humidity of 50% @ 20°C.

Fig 3 reports the dynamic response of NiO nanowire sensor towards acetone and ethanol. The decrease in conductance during the interaction with these reducing gases was observed, which is the typical behaviour of p-type metal oxide sensors.

The sensing response of NiO nanowire sensor was shown in Fig 4(a) with respect to the different temperatures, ranging from 200°C-500°C. From these observations, the optimal working temperature was found to be 500°C for both gases. The calibration curves have been drawn in Fig 4 (b) it was observed that the sensors respond in very similar way towards acetone and ethanol. Even the slope of the calibrations curves for both gases was nearly the same. These curves were fitted by the typical power trend law for metal oxide sensors, and the calculated detection limits resulted in 2.7 ppm for acetone and 2.5 ppm for ethanol.

4. Conclusions

In conclusion, the NiO nanowires were synthesized with VLS technique and were found to be densely packed. The sensitivity of the sensor was tested to different gases and at different working temperatures. The optimum working temperature was found to be 500° C for both gases at relative humidity of 50%. The nanowire sensor response toward both acetone and ethanol was found to be very similar. From these interesting results, the sensitivity of these sensors for other reducing and oxidizing gases are under investigation.

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