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V_2O_5 Nanorods as CO_2 Gas Sensing Devices

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ABSTRACT

In the present paper, a gas sensing device based on Vanadium oxide thin films (V_2O_5)/ Porous Si (PS) / Si structure has been used to detect CO_2 gas at different concentration. Amorphous and crystalline vanadium pentoxide (V_2O_5) thin films were grown onto monocristalline silicon and porous silicon substrates using the Dip-coating method. The Vanadium oxide has been produced from vanadium alcoxide precursor. Different structures based on V_2O_5 / Porous Si/ Si have been realized and studied. Current-voltage (I-V) characteristics show that the sensor properties were modified due to CO_2 gas presence. The structure sensitivity increases potential and concentration of CO_2 increase. In addition, the structure exhibits fast response and 32s recovery time. The Obtained results are promising since the measured response and recovery time were lowered compared to CH/PS/Si structure.

INTRODUCTION

The need to control air from pollution in our environment, in laboratories, hospitals or generic technical installations, pushes to the development of highly sensitive gas detectors in order to prevent accidents caused by gas leakages.

Ideal gas sensors should have: high sensitivity, high selectivity, negligible response to other species, small size, and low cost.

In the last years, new sensing materials, apart from the standard thick film oxides, have been considered. Because of the very large surface/volume ratio, high reactivity, and the potential compatibility with silicon-based electronics, porous silicon (PS) is one of the most promising materials for gas sensors fabrication. Indeed PS-based devices have been proposed as sensors for humidity [1,2], NO_x [3,4] and various organic polar substances [5,6]. The involved sensing mechanisms are generally associated with the free carrier concentration change in the porous layer due to adsorbed molecules, or changes in the dielectric constant due to gas condensation inside the pores; the sensed quantity is generally conductance to current [7] or capacitance [2,8]. PS has been used for gas detection at relatively low temperatures, even at room temperature, compared to other MOS gas sensors, PS produced by silicon can be fully integrated into the silicon microchip. But the exploitation of the PS based gas sensing is very limited by its instability of sensitivity and lack of thermal stability [9].

To enhance performances of the sensors and reduce the working temperature, structure of metal oxide/PS is formed. In the last years some works on the gas sensitivity of PS/WO3 [10], PS/ZnO composites [9,11]. Very recently, the PS/ V_2O_5 structures have been reported in the literature and studied such as highly sensitive ethanol vapors [12] and Ammonia sensor material [13].

Most widely studied gas sensor components today are probably metal-oxide-semiconductor (MOS gas sensors. MOS sensors are a strong candidate for different types of gas sensing systems. They have a simple operating principle in the contact of the gas, low cost, and high sensitivity. Vanadium oxides are one candidate for metal-oxide gas sensor.

Vanadium pentoxide (V_2O_5) is one of the most stable metal-oxide semiconductors in the vanadium-oxygen system, with an

energy gap of 2.2~2.5 eV [14-16]. It is well known that the optical and electrical properties of V_2O_5 films are different from those of the corresponding bulk materials [17,18]. The properties of amorphous, microcrystalline, and nanocrystalline V_2O_5 films, such as the crystalline fraction, grain size, and crystal orientation are highly dependent on the microstructure [19,20].

Various nanostructures of V_2O_5, such as nanorods, nanowires, nanobelts and nanotubes, have been considered for application in many industrials technological applications, such as heterogeneous catalyst [21]. V_2O_5 is an active electrode in a rechargeable lithium battery [22], indeed high electrochemical activity, high stability and thin film ease formation by numerous deposition techniques led to its use as a highly promising intercalation material in solid state micro-battery applications. However, stoichiometric vanadium oxide films are difficult to be obtained [23].

Because nanostructured-V_2O_5 has interesting properties that are very different from those of thin-film and bulk V_2O_5, numerous effective approaches to grow nanostructures have been developed [24-27]. There are several methods for growing V_2O_5 nanostructures such as RF sputtering [28,29], magnetron sputtering [30] flash evaporation, sol-gel technique [31] and pulsed laser deposition [32]. However, technique with relatively low cost and large area deposition as the dip-coating has been chosen in this work to prepare this material.

In the present work, the nanostructured vanadium oxide thin films have been synthesized by using a sol-gel process from vanadium alcoxide precursor, and the final gas sensing device based on Vanadium (V_2O_5)/ PS /Si structure has been used to detect CO_2 gas at different concentration, and at room temperature.

EXPERIMENTAL

PS layers were grown by electrochemical dissolution in an HF-based solution on a single- crystalline p-type (100) heavily-doped Si substrate. Substrate nominal resistivity was 1-10 Ω cm, and has 450 µm thicknesses. Before the anodization, the native oxide was removed from the wafers backside, and aluminum back contacts were deposited with In/Ga mixture. The anodizing solution was obtained by mixing 49% aqueous HF solution with ethanol in a volumetric ratio 1:1. Different solutions were tested; the etching was performed with 20 and 50 mA/cm^2 during 5 minutes. The samples where then rinsed in ethanol and hexane and dried in N_2 environment. The vanadium pentoxide thin films have been prepared by dip-coating using vanadium (V) oxytriisopropoxide ($VO(OPr^i)_3$) (Sigma Aldrich) with isopropanol (Pr^iOH)) in presence of acetyl acetate. The PS substrate was dipped into the above prepared solution for 1min per cycle. Three dipping cycles were performed for each sample. The deposited samples were then annealed at different temperatures. The V_2O_5 thin film crystallized at 500°C.

In order to collect a maximum of electrical current, Aluminium (Al) electrode is deposited onto the surface of samples in form of frame of 1 mm using vacuum evaporation method. **Figure 1** illustrates the realized gas sensor device. The obtained samples were characterized by various techniques including surface morphology and microstructure, investigated by using JEOL JSM 6360 LV scanning Electron Microscope (SEM). X-raybdiffraction (XRD, Bruker D8 Advance), Voltamperometric is employed for the sensor electrical characterizations in a controlled gas environment.

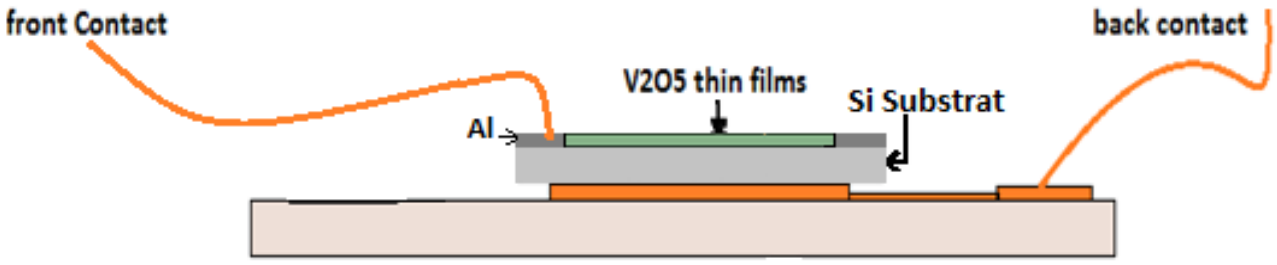

Figure 1. A schematic diagram of the V_2O_5/PS/Si sensor.

RESULTS AND DISCUSSION

Microstructure Analysis

Figure 2a present the SEM image of the as – deposited vanadium, granular homogenous film dipped on the substrate. A drastic change of morphology occurred after annealing at 500°C in air.

Long striped of V_2O_5 Nano rods with widths about 80-100 nm and lengths of 1-10 µm where synthetized as shown in **Figures 2b and 2c**.

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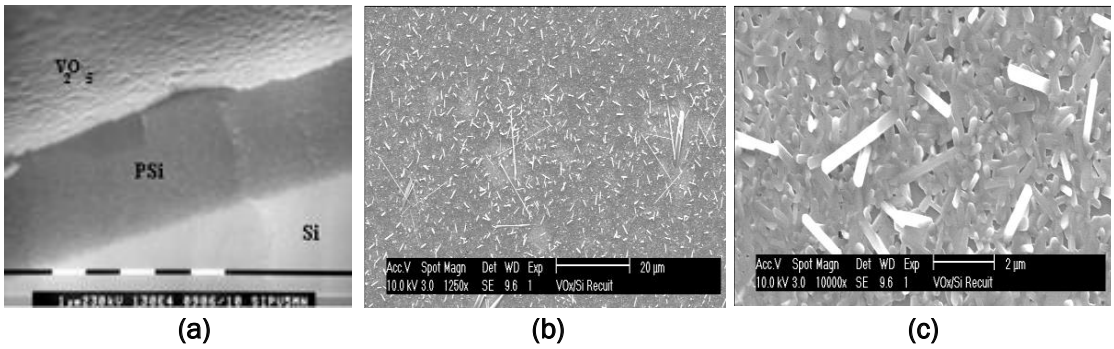

Figure 2. SEM image of deposited vanadium film by dip coating technique, (a) as deposited vanadium, (b) after annealing at 500°C in air, (c) Magnification of (b).

The XRD Characteristics

XRD analysis was carried out to identify the crystalline structure of the V_2O_5 nanorods. In XRD spectrum presented in **Figure 3**, diffraction peaks can be indexed to orthorhombic V_2O_5 phase. The peak corresponding to the V_2O_5 (010) appeared at 2θ=20.38°.

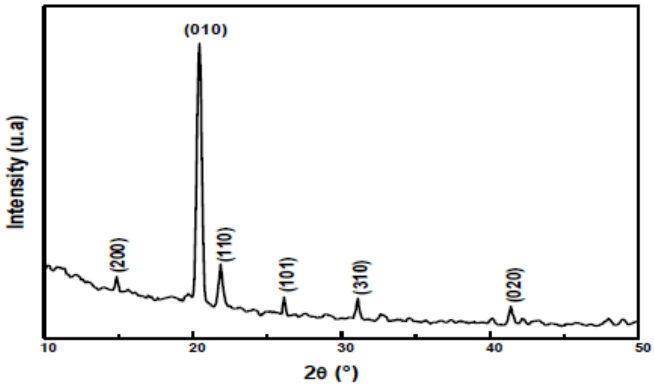

Figure 3. XRD spectrum of V_2O_5 nanorods.

The Current Voltage (I-V) Characteristics

The Al/ V_2O_5/PS/Si structure current voltage characteristics were measured in air environment and under CO_2 gas with different concentration from 0.5 to 4 mbar. In **Figure 4** that I-V curve shape does not change under CO_2 gas exposure. However, a change is observed in current magnitude. At 0.5 V fixed potential, we notice 11 µA current variation, before and after 1 mbar CO_2 exposure whereas 22 µA variation is recorded at 1 V potential.

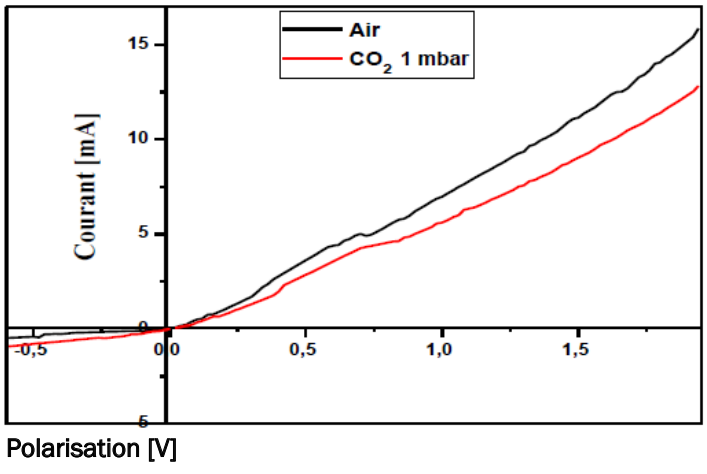

Figure 4. I-V structure characteristics for 1 mbar CO_2 gas.

The Current Variation ($\Delta I = II_g - I_a I$)

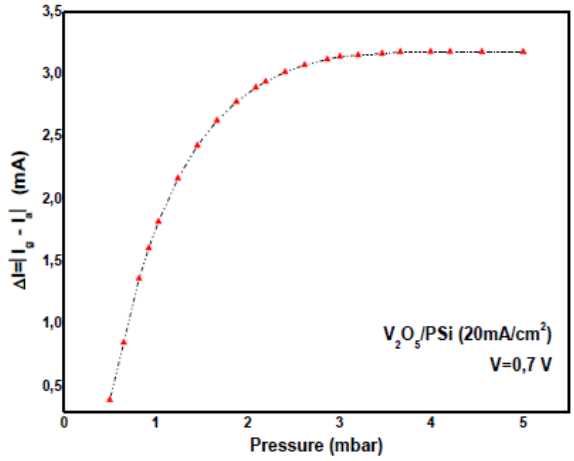

Figure 5. Current variation ($\Delta I = II_g - I_a I$) versus CO_2 gas pressure.

Figure 5 depicts the current variation ΔI versus CO_2 gas pressure, where I_g and I_a (or I_{gas} and I_{Air}) are respectively the currents acquired in air condition and after gas contact. The CO_2 adsorption modifies the resistivity at the V_2O_5/PS surface structure. In addition, **Figure 5** shows clearly that the current variation increases with gas pressure, reaches a maximum around 2.8 mbar pressure, and then becomes constant. It is interesting to notice that the same shape variation has been observed for sensors based on CH_x-PS/Si structure in CO_2 and H_2 gas presence [33]. However, for CO_2 gas constant concentration, the sensor based V_2O_5/PS structure current variation is found lower than that obtained for CH_3/PS- based sensor. This may be attributed to vanadium oxide layers presence [34].

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The Sensitivity Measurement ($\Delta I/I_a = II_g - I_a I/I_a$)

Figure 6 shows the sensor sensitivity as function of polarisation, for different concentration of CO_2 gas. It shows that the structure sensitivity increases with the increase of bias potential. It reaches about 50% maximum sensitivity for 0.08 V, then decreases and stabilizes at 20% sensitivity for potential polarisation higher than 0.1 V. And we observe the repeatability of this result for different pressures of CO_2 gas. We made further measurements on different samples and different CO_2 pressures, to confirm these results. This result indicates that the proposed detection device can work at low energy consumption.

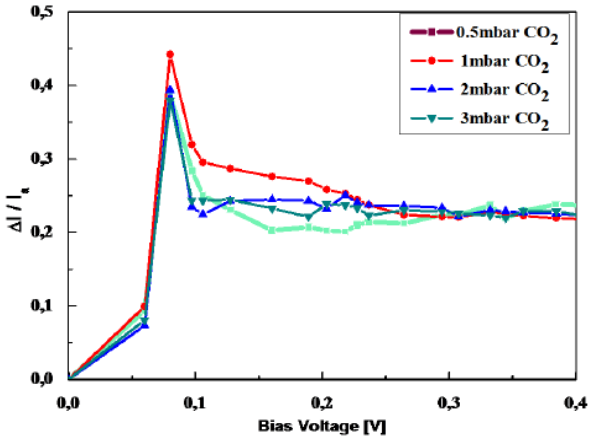

Figure 6. Sensor sensitivity ($\Delta I/I_a$) versus bias voltage for 1 mbar CO_2.

The dynamic response of the structure for a CO_2 concentration of 1mbar at +1V, at room temperature, is depicted in **Figure 7**. It shows a large current variation 1.2 mA for the tested CO_2concentration. The phenomenon is reversible when the gas is removed, and the current recovers its initial value. Values of 30 and 25 second were found for response and recovery time, respectively. In addition this this realized sensor, indicate a fast response in CO_2 gas presence, the response time is found quasi-similar to the recovery time with the same adsorption rate than for gas desorption from the structure surface. These results are very interesting since the measured response and recovery times are lower than those generally observed for CH_x/PS/Si structures against CO_2 gas [34].

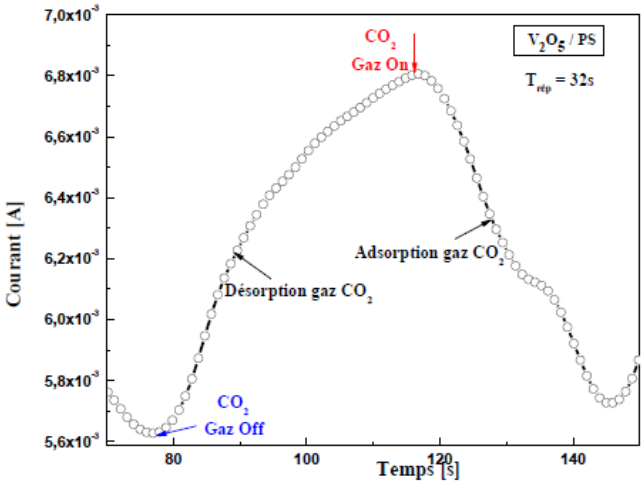

Figure 7. V_2O_5/PS/Si sensor dynamic response to 1 mbar CO_2 gas concentration and +1 V applied voltage.

CONCLUSION

Gas sensing device based on Vanadium oxide thin films (V_2O_5)/ Porous Si (PS) / Si structure has been used to detect CO_2 gas at different concentrations. Amorphous and crystalline vanadium pentoxide (V_2O_5) thin films were grown onto monocristalline silicon and porous silicon substrates using the Dip-coating method. Current voltage characterization shows that the sensor characteristics are modified with the gas presence. The structure sensitivity increases with bias potential increase and CO_2 concentration. In addition, the structure exhibits fast response and recovery time around 30s. By mean of consequence, the proposed structure can operate at low voltage in the interval 50 to 100 mV.

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