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On the sensing properties of Nb_2O_5 films prepared by pulsed laser deposition

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***Abstract*—This paper reports the electrical characterization of the sensing properties of nanostructured Nb_2O_5 films deposited by Pulsed Laser Deposition (PLD) towards ethanol gas. In particular, a sensing thin film was deposited by 2000 pulses of Nd:YAG UV laser (operating at λ = 266 nm and energy of 112 mJ) obtaining a porous layer composed by vertical nano-pillars with a thickness of about 500 nm. The sensing film was characterized in a temperature operating range from 340 °C to 450 °C, identifying the optimal working temperature at 420 °C. Sensing response toward ethanol was determined in a concentration range from 50 ppm to 100 ppm. Response repeatability, stability, reversibility and recovery times were tested as well. The preliminary characterization highlighted the very promising sensing performance of PLD niobium oxide thin films. Therefore, the investigation of ethanol gas sensors based on PLD sensing layers grown in different conditions to tune nanostructure and thickness, is in progress in order to further increase the sensor sensitivity and selectivity.**

Keywords*—*Pulsed laser deposition, MOX gas sensors, ethanol sensor, Nb_2O_5

I. Introduction

During these years, nanostructured sensing materials have been the turning point in the development of gas sensors based on metal oxide semiconductors, empowering the technological solutions in terms of developed devices, high performance and number of applications [1, 2]. From the side of sensing material development, there are many techniques reported in the literature, spanning from chemical preparations like sol-gel synthesis [3] to physics-related ones like sputtering [4], laser ablation and pulsed laser deposition [5, 6]. This paper reports the preliminary characterization of sensing properties towards ethanol of Nb_2O_5 films prepared by Pulsed Laser Deposition (PLD).

Pulsed Laser Deposition is a very promising deposition method widely applied in recent years to deposit nanomaterials for sensing applications [7, 8, 9, 10]. Typically, depositions occur inside a vacuum chamber that, optionally, can contain reactive gases at low pressure. A high-power pulsed laser is used to vaporize the target material by means of extremely short (few nanoseconds) focused laser pulses. The vaporized material expands in the deposition chamber reacting with the substrate where it deposits in the form of a nanostructured thin film whose properties and morphology largely depend on deposition conditions, laser pulse characteristics, substrate and target material. If there is any reactive gas in the chamber during the deposition, the target material, after vaporization, may react with the gas, depositing on the substrate as some specific compound. As an example, oxygen is typically introduced in the deposition chamber to deposit metal oxides starting from raw metallic targets.

Several conductometric sensors have been manufactured by using PLD technique due to the good electrical, chemical and morphological properties of the deposited films [11, 12]. Conductometric gas sensors based on metal-oxide semiconductors are nowadays one of the most studied sensing devices in the field of gas sensors thanks to their quite low cost, very promising performance and relatively simple readout requirements. The working principle of such sensors is based on the capability of metal-oxide semiconductor films to have their conductivity modulated by the presence of some target gas. Typically, the surface of such sensing film has a high concentration of superficial defects which behave as

absorption sites for gas molecules. This is especially true for nanostructured films in which the surface-to-volume ratio can be very large. The presence of such defects, which often bond the oxygen present in the air, involves the creation of depletion/enrichment layers within the sensing film. As a consequence of such layers, the conductivity of the film changes. When a target gas is present, a reaction occurs between the gas itself and the available free sites and-or the chemisorbed oxygen, with a consequent variation in the thickness of the charge layers. The variation of the depletion/enrichment layers involves a modulation of the film conductivity which is related to the concentration of the target gas.

The sensing performance of such devices mainly depends on the film chemical composition, structure and morphology. Moreover, film temperature plays a very important role being chemisorption/desorption/diffusion processes strongly related to the temperature. Therefore, typically a specific temperature exists, at which the sensor sensitivity is maximized for a specific target gas. Most of the recent research in this field is focused on improving the sensitivity, selectivity and stability of such sensors, trying at the same time to decrease the operating temperature in order to reduce the power consumption.

Niobium oxide is a promising sensing material which behaves as an N-Type semiconductor. As a consequence, either the chemisorption of air oxygen or the superficial defects develop a depletion layer in the film with consequent increase of the film resistance. When the target gas reacts on the surface, the bonds are broken and the electrons return in the conduction band (depletion layer is reduced) with a reduction of the film resistance which is related to the concentration of the target gas. The response of such a sensor is typically given in terms of film resistance variation with respect to pure air condition:

$$S = \frac{R_0}{R} \qquad (1)$$

where R_0 is the resistance of the film in pure dry air, and R is the resistance under a specified concentration of the target gas.

In literature, Nb_2O_5 has been employed to develop several conductometric gas sensors, especially for ethanol and acetone [4, 13, 14], with quite good sensing performance, different concentration range and selectivity, depending on the deposition method and the characteristics of the nanostructured film. In [15], the Authors tested the performance of an ethanol sensor based on stoichiometric solid solutions of Sn, Ti and Nb oxides prepared by sol-gel method. Specifically, they investigated the effect of humidity on the sensors' response, finding the beneficial effect of Ti which weakens the interaction with water and facilitates the desorption of physisorbed H_2O molecules at high operating temperature.

In this work, the possibility of developing an effective conductometric gas sensor is explored by depositing a sensing Nb_2O_5 thin film by means of Pulsed Laser Deposition on a tiny alumina substrate. In particular, the manuscript presents the characterization of the sensing film including operative temperature, sensitivity, stability, response and recovery times.

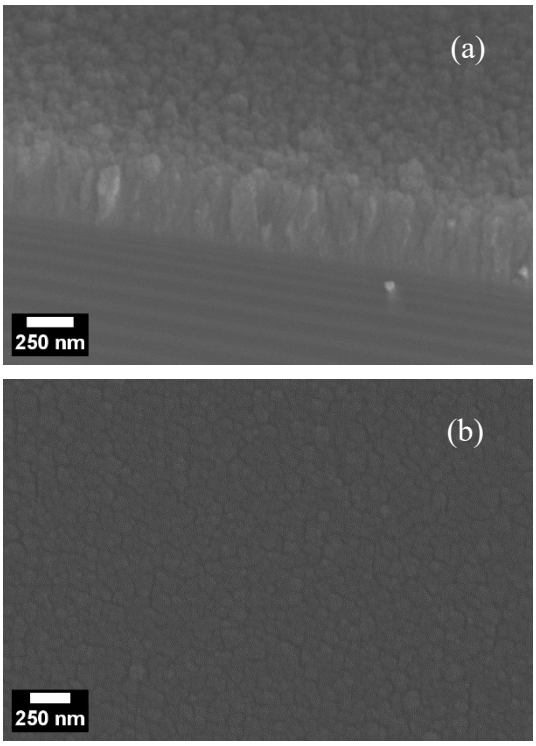

Fig. 1. SEM micrographs of the Nb_2O_5 sensing film deposited by Pulsed Laser Deposition (2000 pulses). Cross section (a) and top (b) view.

II. The Sensing Material

The sensing film was prepared by Pulsed Laser Deposition (PLD). A Nd:YAG UV laser operating at λ = 266 nm with pulse duration of about 4 ns is focused on a target made from the source material (Nb_2O_5). The oxide deposition is performed in a vacuum chamber (containing the target material and the alumina substrate) at the pressure value of $5 \cdot 10^{-5}$ Pa. Then pure oxygen is introduced in the chamber until reaching a pressure between 10 Pa and 20 Pa. The target was prepared by hydraulic pressing the pure powder obtained from Onyxmet company (Nb_2O_5, purity 99.9 %), then the pellet was sintered by thermal treatment in a furnace at a temperature of 1200 °C for 8 hours. The Nb_2O_5 thin film characterized in this paper was deposited in the above conditions with a sequence of 2000 laser pulses generated by Lumibird Q-Smart laser equipped with 4th harmonic generator (266 nm), average energy per pulse of 112 mJ. Fig. 1 (a, b) shows the Scanning Electron Microscope images (recorded using a FESEM Zeiss Supra 40 instrument, with an accelerating voltage of 10 kV) of the Nb_2O_5 thin film. A rough nanostructured pillar morphology, suitable for gas sensing applications, was observed. In particular, pillars have an average diameter of about 70 nm, oriented vertically one next to the other and leaving a rough porous surface which is expected to increase the surface-to-volume ratio (with respect to a bulk material) and, therefore, improve the film sensitivity to gaseous compounds.

III. Experiments

The sensing film was deposited on some alumina substrates equipped with a couple of platinum interdigitated electrodes on one side and a platinum serpentine on the opposite one. The film was deposited on the interdigitated electrodes, while the serpentine was biased during characterization of the film in order to set a proper operating

temperature. The interdigitated electrodes are in parallel configuration, in this way, the electrical resistance, exposed within each couple of fingers is in parallel with the ones beside. This configuration is suited for sensing films with high resistive values, bringing to lower, and average values related to the number of fingers composing the interdigitated path. The alumina substrates have a rectangular shape of 3 mm × 6 mm and a thickness of about 1 mm. On the side with interdigitated electrodes there are three fingers for each contact (i.e. 5 couples and, mainly, 5 electrical resistance in parallel, as shown in Fig. 2), characterized by a width of 199 µm ± 15 µm, a spacing of 198 µm ± 11 µm and a thickness of 7.2 µm ± 0.8 µm (with a nominal value of pitch and thickness of 200 µm and 10 µm, respectively). On the opposite side, the heater shows a mean platinum thickness of 7.6 µm ± 0.6 µm, a width of 212 µm ± 29 µm, and a spacing of 206 µm ± 12 µm (same nominal values of the other side). In both cases, the first number represents the average value, and the second indicates the standard deviation computed from profilometer data coming from the dashed path in Fig. 2. Measurements were taken from six fingers of the interdigitated electrodes and six traces of the serpentine-shaped heater, with each path measured five times. The prototype was placed in a Teflon test chamber with a volume value of 5 cm^3, as shown in Fig. 2.

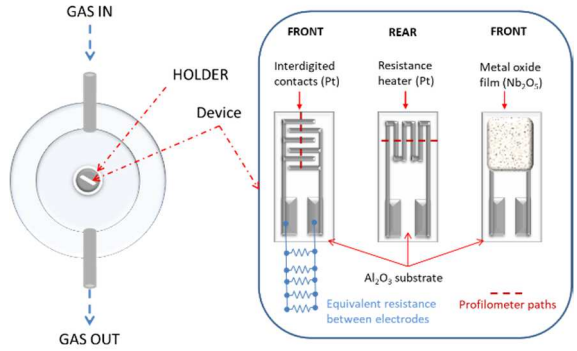

Fig. 2. Schematic of the test chamber and of the sensing prototype.

The characterization protocol was carried out by connecting the test chamber to a measurement system equipped with an array of gas pipelines; each line has a certified gas bottle, a mass-flow controller and some of them can mount a permeation tube. In this way, it is possible to span for several concentration values and for different gas target typologies. The electrical test system is composed of a Keithley 2400 Source Measurement Unit (SMU) to record the resistive value of the film when exposed to a specific gas concentration and of a Keysight 36313A Power Supply able to bias the platinum serpentine, in order to set the proper operating temperature value. The system is able to expose the sensing film to a maximum flow value of 100 cm^3/min, the different values of ethanol concentration are reached by means of a certified permeation tube provided by Fine Permeation Tubes (permeation rate of 9740 ng/min ± 5%) set to an operating temperature value of 40 °C and by varying the flow value carried in the pipeline. The total flow value changes with the target gas concentration. However, it is kept constant for a specified target gas concentration when switching from the baseline (dry air) to the gas target (mixture of dry air and ethanol) and backwards without any change of dynamic conditions of the gas flowing into the chamber.

IV. RESULTS

In order to evaluate an optimal temperature operating value, a characterization of the sensing layer in a gaseous concentration of 50 ppm of ethanol vs. temperature, in the range from 340 °C to 450 °C, was performed. The results are shown in Fig. 3. It is possible to observe a bell shape behavior, well known in the literature, with a maximum response value at 420 °C. When exposed to ethanol, the value R of the electrical resistance decreases with respect to the value recorded in dry air conditions (R_0). For this reason, the response is evaluated as the ratio between the resistance value at the baseline (dry air) R_0 and the gas target one R.

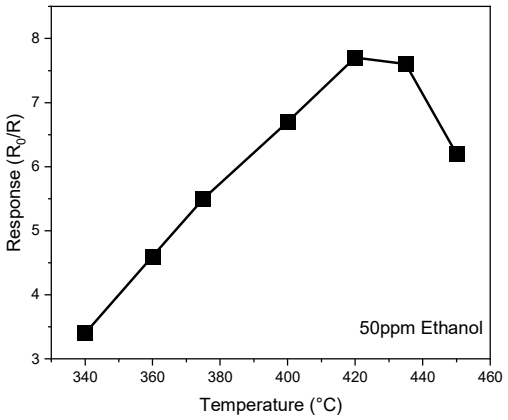

Fig. 3. Response at 50 ppm of ethanol vs. temperature. Optimal working temperature for the sensing film is of 420 °C.

Repeatability tests were carried out at several ethanol concentration values. In Fig. 4, tests at 50 ppm (a) and at 75 ppm (b), are reported.

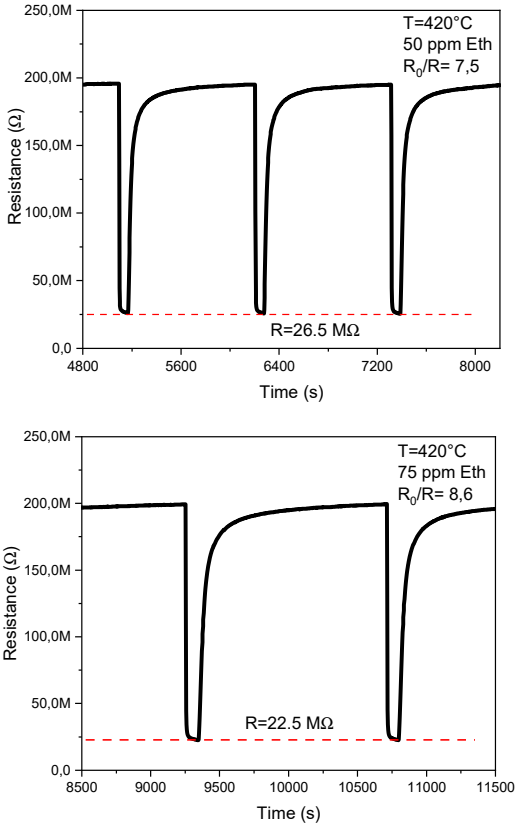

Fig. 4. Repeatability test at 50 ppm (top) and 75 ppm (bottom) of ethanol.

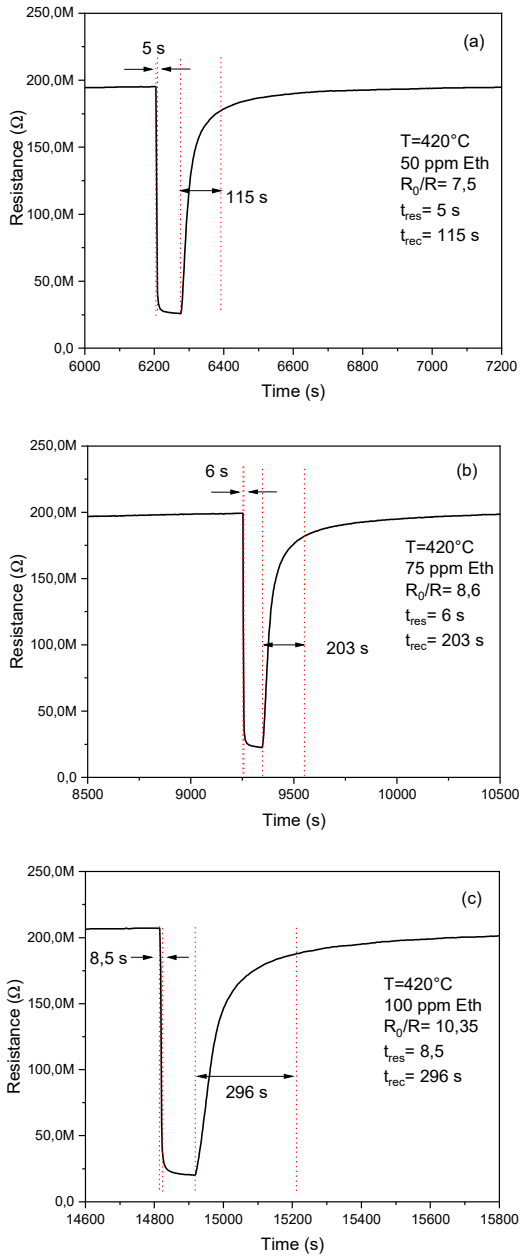

Fig. 5. Response and recovery time values at 50 ppm (a), 75 ppm (b) and 100 ppm (c) of ethanol and at 420 °C.

Such tests were performed by switching from baseline to gas target value. It can be seen how the sensor sensing layer reaches the baseline and the saturation gas target values with very good stability. These results brought the research activity to evaluate also the dynamic performance in terms of response and recovery time values. Since the gas flow varies from a minimum of 50 cm^3/min (for 100 ppm concentration value) to a maximum of 100 cm^3/min (for 50 ppm concentration value), the time spent to evacuate the volume of the chamber (5 cm^3) can be neglected.

In Fig. 5 are reported the response and the recovery time values of the sample when exposed at several ethanol concentration values (a, b, c). The response time is defined as the time interval needed to pass from the baseline value to the 90% of the target saturation value. The recovery time is defined as the time interval needed to pass from the target saturation value to the 90% of the baseline value. The reported plots show fast response and recovery time values and very good reversibility. The response and recovery times were also evaluated in the whole temperature range, as reported in Fig. 6. The measurement protocol was to keep the gas concentration constant and to switch between baseline and target gas at several temperature values (once reached the thermal stability). It is possible to observe a slight negligible variation of the value of the recovery time reported in Fig. 5 (a) (115 s and 123 s, respectively), probably due to slightly different conditions of the measurement protocols (the first by variating concentration and the second one by variating the temperature).

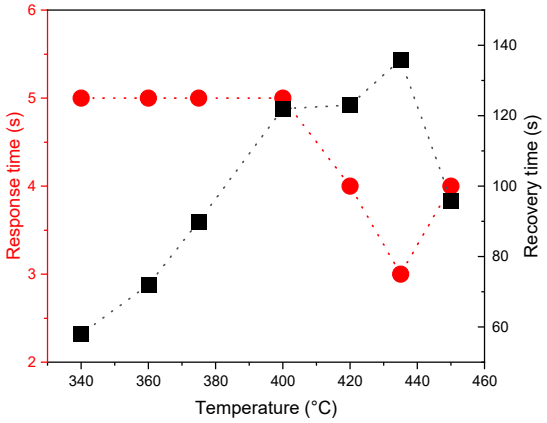

Fig. 6. Response and recovery time values at 50 ppm as a function of temperature.

The sensing properties of the film towards ethanol are also clear in Fig. 7, where the response of the sample is reported as a function of ethanol concentration at 420 °C. The linear behavior is also described with good agreement with the linear interpolation also reported in the figure.

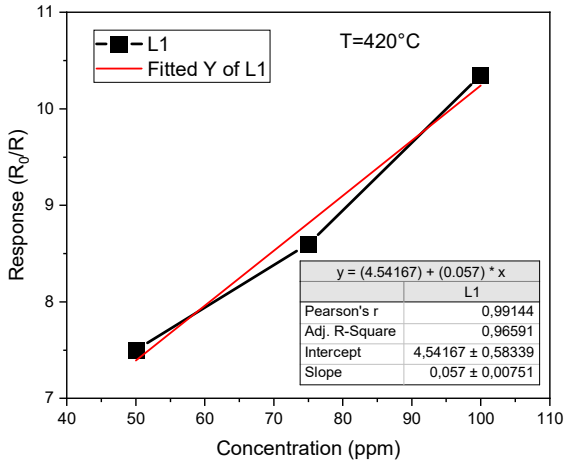

Fig. 7. Sensor response as a function of ethanol concentration.

V. CONCLUSIONS

In this paper the preliminary test of the sensing properties towards ethanol of Nb_2O_5 films prepared by Pulsed Laser Deposition (PLD) are reported. In particular, tests regarding short term repeatability, reversibility and response/recovery times showed good and promising results. Therefore, with the aim of developing a reliable chemoresistive gas sensor, further activities are in progress to evaluate other important sensing performance such as selectivity and long-term stability which

are critical aspects for this type of sensors due to the well-known effects of sensing film contamination and degradation.

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