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Employment of Nb_2O_5 thin-films for ethanol sensing

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***Abstract*—Gas sensors attract the attention of research since years because of their good performance and the extremely wide field of application. Environmental monitoring, chemical and industrial processes, automotive, medical diagnosis and healthcare are few of such applications. Chemical gas sensors are able to detect specific gases in a wide range of concentration going from few ppb to hundreds ppm. This paper deals with such a topic and presents a novel gas sensor for ethanol based on a Nb_2O_5 thin sensing film. The sensor was realized by depositing the niobium oxide layer on a tiny alumina substrate by means of a lab-scale plasma sputtering reactor. Subsequently, the sensor was characterized and it revealed good sensing performance towards ethanol. Ethanol monitoring finds application in several fields including breath analyzers for assessing drunk-driving situations, industrial monitoring, medical diagnosis and environmental safety. The proposed sensor, thanks to its good sensitivity, selectivity, wide concentration range, low-cost and low power consumption, can be a suitable solution in such applications.**

***Index Terms*—Gas sensors, ethanol sensing, niobium oxide**

I. Introduction

Nowadays, the demanding of high performing gas sensors is constantly growing. Environmental monitoring for detection of pollution and harmful gases, chemical and industrial processes, medical diagnosis and breath analysis, security and food quality tests are few cases where gas sensors find wide application.

Ethanol is an example of target gas whose monitoring is required in many applications going from safety and explosion prevention, bio-pharmaceutical manufacturing, chemical and paint industry to breath analysis and drunk-driving test. Therefore, the availability of effective, low-cost and low power consumption ethanol gas sensors [1], [2], [3] is extremely important, especially in applications which require small and portable sensing devices.

Several different approaches have been exploited for the detection of gases and among them there are optical sensors [4], [5], [6], [7], electrochemical sensors [8], [9], acoustic devices [10] and micro-wave transduction [11]. However, conductometric ones are surely the most studied due of their sensing characteristics such as high sensitivity, low power consumption, small size, low-cost and easiness in the read-out [12].

Conductometric gas sensors are sensing devices which change their conductivity as function of the concentration of a specific gas. Such a type of sensors is basically implemented by two electrodes which perform an electrical contact with a sensing material. Nowadays, several sensing materials have been investigated including metal oxides, carbon-based nano-materials, such as graphene and carbon nanotubes, and polymers [13], [14]. Among all these materials, metal oxides are probably the ones which attracted the most interest and they are therefore the most investigated.

Metal-oxide gas sensors (MOX) base their working on superficial vacancies and broken bonds which acts as iono-sorption centres for the gas molecules present in the environment. Such gas molecules easily bond on the sensing material surface with the consequent trapping of a specific amount of electrons. As a consequence of such a mechanism the conductivity of the sensing material change according to the gas concentration [15], [12], [16].

A dedicated read-out system can measure variation of the sensor resistance in presence of the target gas and, from such a value, it is possible to determine the sensor response defined as:

$$S = \frac{R_a}{R_g} \tag{1}$$

where R_a is the reference resistance (typically measured in pure air) and R_g is the sensor resistance in presence of the target gas.

In general, the surface bonding mechanism is competitive among the different gaseous species present in the environment. Typically, oxygen is always bound on the surface and, when other species are present, they can either chemically react with the adsorbed oxygen or directly replace oxygen in the bonds. Such process are controlled by several factors such as chemical affinity, diffusion processes, thermal energy [17]. In particular, temperature is a critical parameter in the working of such sensors because almost all such factors are temperature-dependent with the best operating temperature between room temperature and few hundreds Celsius degrees according to the sensing material and the target gas. Therefore, the read-out system has to provide a way to accurately control the sensor working temperature in order to assure a reliable measurement.

Many works deal with this topic and several different metal oxides, often mixed with noble metals, have been investigated in term of their sensing performance towards many target gases. However, there are some interesting oxides whose capabilities are not still fully assessed. Niobium pentoxide is one of them, although few works highlight its promising sensing characteristics dealing with target gases such as oxygen, hydrogen, carbon monoxide and ammonia [18], [19], [20]. However, the possibility to employ pure niobium oxide for detecting ethanol at the ppm level has not been still investigated.

Therefore, this paper investigates the sensing performance of niobium pentoxide (Nb_2O_5) towards ethanol by fully characterizing a sensor prototype realized by depositing a $200\ \mathrm{nm}$ Nb_2O_5 sensing film over a tiny alumina substrate. The characterization tests are described in detail and the achieved results discussed. Such a material, which already demonstrated excellent sensing performance towards acetone [21], revealed quite good characteristics also in the sensing of ethanol making it suitable in several applications.

II. Material Deposition and Characterization

The proposed sensor was realized by depositing a thin film of Nb_2O_5 over a small alumina substrate whose dimensions are $3\ \mathrm{mm} \times 6\ \mathrm{mm}$ with a thickness of about $1\ \mathrm{mm}$. The small substrates are available off-the-shelf in slices of 98 tiny units. Each substrate features a small platinum heater on the back face and a couple of platinum interdigitated electrodes on the top face. Fig. 1 shows the slice and the detail of a single substrate with the sensing film already deposited over the electrode area.

The small platinum heater is employed to control the sensor working temperature and it is characterized by a typical resistance of $4.5\ \Omega$. A power of about $1.2\ \mathrm{W}$ is suitable to reach a working temperature around $400\ ^\circ\mathrm{C}$. The interdigitated electrodes, instead, perform an electrical connection to the deposited sensing film and, at the same time, the interdigitated structure helps to decrease the effective sensor resistance by paralleling the small areas of the sensing film. The substrate provides four small platinum contacts which allow one to connect the sensor to the read-out system by using a small ceramic connector.

The Nb_2O_5 film was deposited by reactive plasma sputtering in a lab-scale capacitively coupled parallel-plate reactor with an asymmetric electrode configuration working at a RF frequency of $13.56\ \mathrm{MHz}$. Depositions were performed in a mixture of argon and oxygen (70% Ar, 30% O_2) at a power of $250\ \mathrm{W}$ and with a deposition time of $30\ \mathrm{min}$ in the same experimental setup already employed for the deposition of other sensors based on niobium oxide [21]. A niobium target (99% purity) was employed as cathode in order to deposit niobium oxide.

The alumina substrate together with a small silicon reference sample was placed on the ground electrode taking care to cover the substrate contacts with a small alumina mask. The block diagram of the plasma reactor and the arrangement of the substrate inside the deposition chamber are shown in Fig. 2, respectively on the left and right.

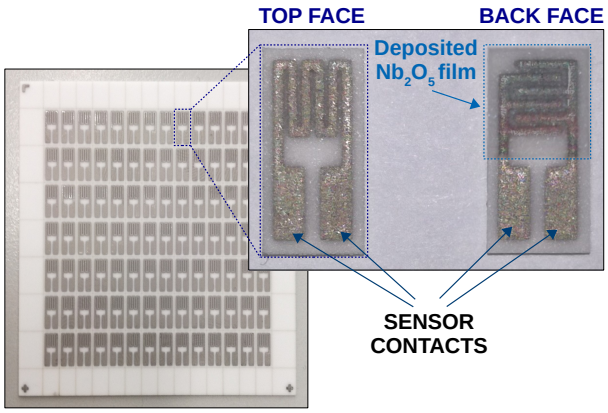

Fig. 1. Slice of 98 substrates and details of a deposited substrate showing top and back faces.

After the deposition, the deposited film was characterized by using the silicon reference sample with results similar to the ones achieved with other Nb_2O_5 sensing films [21], [22]. In particular, a morphological characterization was carried out with a field-emission scanning electron microscope (ZEISS SUPRA 40). Such characterization revealed a compact film featuring a thickness of about $200\ \mathrm{nm}$. A chemical characterization was eventually carried out by X-ray photoelectron spectroscopy (XPS) with the aim of assessing the film chemical composition. The analysis demonstrated that film composition is niobium(V) pentoxide (Nb_2O_5), as expected.

III. Sensing Performance

Several tests were carried out with the aim of assessing the sensing performance of the deposited Nb_2O_5 film towards ethanol. Such tests were carried out by using a dedicated measurement workbench specifically designed for characterizing gas sensors. The workbench, shown in Fig. 3, features a slot of five mass flow meters (BRONKHORST, model: F-201C-FAC-22) which control the gas flow rates from a set of certified gas bottle to the sampling chamber in order to achieve a gas mixture suitable for characterizing several features of the gas sensor, including temperature dependence, sensitivity, selectivity and response times. A digital control system is employed to stabilize the sensor working temperature. Such a system employs a digitally-controlled power supply (AGILENT, model: E3632A) in order to control the heating of sensor by using the substrate platinum heater both as heating element and temperature probe. A picoammeter/voltage source (KEITHLEY, model: 6487) is, instead, employed to measure the sensing film resistance. The sensor is mounted inside a small sampling chamber with volume of about $5\ \mathrm{cm}^3$ which allows one to achieve a fast response. The mass flow controller array and a set of dedicated valves allow one to set a suitable gas concentration, to keep a constant flux flowing in to the test chamber ($100\ \mathrm{cm}^3/\mathrm{min}$) and, at the same time, to

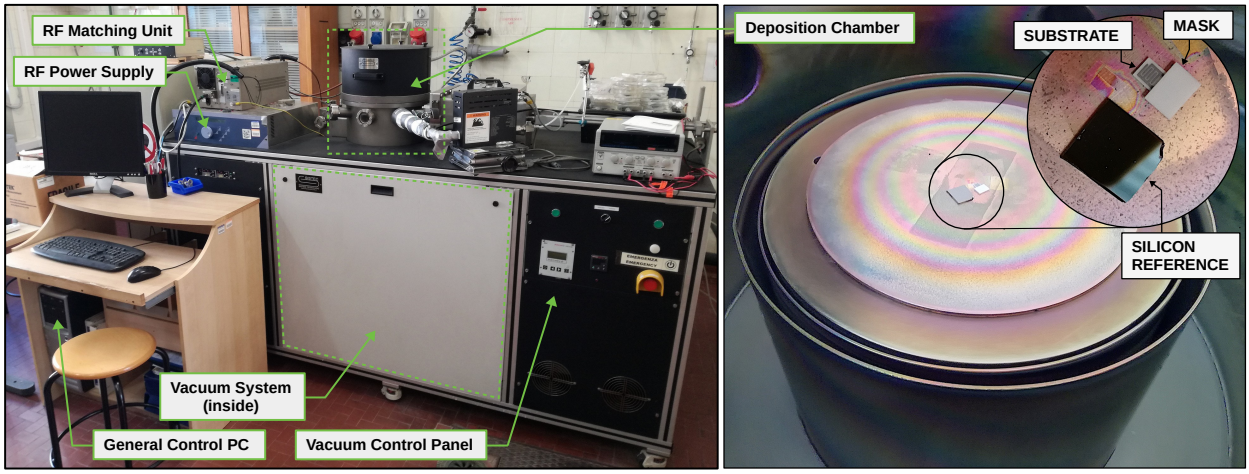

Fig. 2. Plasma sputtering reactor employed in the deposition of the Nb_2O_5 films and detail of the deposition chamber with substrate and reference sample positioned on the ground electrode.

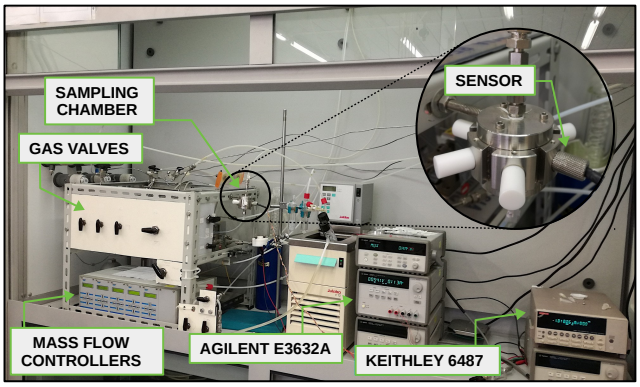

Fig. 3. The measurement workbench employed in the characterization of the sensor performance with a detail of the sampling chamber.

switch between the reference gas (without target gas) and the reference/target gas mixture.

Such a characterization approach, known as a flow-through method, is the one commonly employed for gas sensor characterizations and has several advantages in respect to low-cost devices employing the static-environment method. In particular, the flow-through method allows one to easily and accurately evaluate sensor performance avoiding, at the same time, any possible error due to chemical reactions or adsorption on the chamber walls. In fact, the use of mass flow controllers maintains a constant gas flow rate and gas mass inside the chamber providing to the sensor a steady-state environment which is almost equivalent to a static environment. Moreover, it permits to measure the sensor response times without opening the test chamber and, therefore, avoiding possible contamination due to the external environment. The only possible issue of such an approach is an uncontrolled variation of the sensor working temperature due to the continuous flux which can be avoided by employing a closed-loop control system for the temperature.

A. *Optimal working temperature*

Firstly, a temperature characterization was carried out for determining the optimal sensor working temperature. In particular, the sensor response was measured at different temperatures between $250\ ^\circ\mathrm{C}$ and $400\ ^\circ\mathrm{C}$. A suitable ethanol flow rate was added to synthetic air (80% N_2 and 20% O_2) in order to achieve a fixed concentration of $10\ \mathrm{ppm}$. The ethanol flux was periodically opened by means of the dedicated valve so as to obtain abrupt ethanol peaks and allow one to measure both the reference resistance R_a and the resistance R_g in presence of $10\ \mathrm{ppm}$ of ethanol. The experimental data acquired during such a test are shown in Fig. 4 (left), where it is possible to observe the four peaks in correspondence of the four tested temperatures. In general, sensor response is quite low at $250\ ^\circ\mathrm{C}$ but it quickly increases with the increasing of temperature reaching the best performance at $400\ ^\circ\mathrm{C}$.

Subsequently, sensor relative response as function of working temperature was evaluated by using equation 1. The achieved results are shown in Fig. 4 (right). Here, it is clearly evident how sensitivity increases with the temperature and how optimal working temperature is still not reached at $400\ ^\circ\mathrm{C}$. Unfortunately, the measurement workbench employed does not support temperatures higher than $400\ ^\circ\mathrm{C}$ and, therefore, has not been possible to determine the sensor optimal working temperature. Anyway, it was decided to carry out all the subsequent characterizations at a temperature of $350\ ^\circ\mathrm{C}$ which represents a quite good tradeoff between sensor sensitivity and power consumption.

It is also interesting to observe how the reference resistance changes with temperature going from about $4\ \mathrm{G\Omega}$ at $250\ ^\circ\mathrm{C}$ to about $300\ \mathrm{M\Omega}$ at $400\ ^\circ\mathrm{C}$. This is an important factor because a low reference resistance relaxes the specifications required by the measurement equipment and, at the same time, improves the signal-to-noise ratio of measurements. The reference resistance at the selected working temperature ($350\ ^\circ\mathrm{C}$) is about $700\ \mathrm{M\Omega}$, a value which can be easily

measured by low-cost equipment.

B. Sensitivity towards ethanol

Sensor response towards ethanol was subsequently assessed with a different test. The working temperature was fixed at $350\ ^{\circ}\mathrm{C}$ and synthetic air (80% N_2 and 20% O_2) was employed as carrier gas. Ethanol was properly added to the carrier gas in order to achieve concentration peaks between $10\ \mathrm{ppm}$ and $400\ \mathrm{ppm}$. Fig. 5 (left) shows the experimental data acquired during this test. Subsequently, reached $400\ \mathrm{ppm}$, the ethanol concentration was reduced again to $10\ \mathrm{ppm}$ with the aim to characterize stability and reversibility of the sensor.

The response as function of ethanol concentration was subsequently evaluated by extracting the R_a and R_g values for each peak and by calculating the relative sensor response, with equation 1. Such results are shown in Fig. 5 (right). Here, it is possible to note how the sensor response increases with the ethanol concentration even though the sensitivity slightly decreases. However, no significant sensor saturation is visible within the tested range. Such a sensitivity is quite high and the absence of a significant saturation is a very good characteristic of this sensor.

Furthermore, the test demonstrated a good sensor stability and reversibility. In particular, sensor resistance recovers almost perfectly to the reference resistance R_a of about $756\ \mathrm{M\Omega}$ with a standard deviation of about $4\ \mathrm{M\Omega}$ during all the test. Repeatability of measurement is good as well, being the maximum response variation at the same concentration about 0.07.

A non-linear fitting can be performed in order to determine an experimental mathematical relation between ethanol concentration. Such a relation can be inverted to find a mathematical formula which can be used to calculate the ethanol concentration from the measurement of R_a and R_g.

C. Stability and selectivity

Eventually, other two tests were performed with the aim of assessing the sensor stability and selectivity. The first test was carried out in synthetic air with added a small concentration of ethanol. The ethanol valve was periodically opened and closed in order to generate several repeated ethanol impulses. As an example, Fig. 6 shows the sensor response to five repeated ethanol impulses at $10\ \mathrm{ppm}$. The test revealed a good sensor stability: no significant offset or hysteresis in the sensor response are visible. Acquired data revealed an average reference resistance $R_a = 735\ \mathrm{M\Omega} \pm 20\ \mathrm{M\Omega}$. The relative response of the sensor results very stable with an average value of $S = 2.19 \pm 0.01$.

Also selectivity of the sensor is quite good. The selectivity test was carried out using the same carrier gas (synthetic air) with additions of several possible interferent gases at different concentrations. Tested gases include CO_2, H_2, NH_3, NO_2 and acetone. Sensor sensitivity is negligible towards all the tested gases with the exception of acetone whose response, unfortunately, is higher than ethanol one. Acetone interference can be an issue in some specific applications, such as the drunk-driving breath test. In fact, acetone can be present in the exhaled breath at concentration of few ppm, especially when the subject is affected by diabetes, and such an high sensitivity towards acetone can compromise the reliability of ethanol detection. Therefore, further modifications of the sensor can be investigated in order to improve selectivity to ethanol in respect of acetone. Small addictions of catalytic dopants, such as gold, silver and platinum, is a possible strategy which might improve selectivity and sensitivity. Table I reports a summary of the selectivity tests highlighting the sensor response, normalized by the tested gas concentration, and the correspondent concentrations.

TABLE I
NORMALIZED SENSOR RESPONSE TOWARDS SEVERAL POSSIBLE INTERFERENT GASES.

Gas	Concentration (ppm)	Normalized response
ETHANOL	5 ppm	0.32
ACETONE	5 ppm	0.8
CO_2	500 ppm	0.01
H_2	10 ppm	0.1
H_2	80 ppm	0.02
NH_3	200 ppm	0.015
NO_2	5 ppm	0.02

IV. CONCLUSIONS

The paper deals with the characterization of the sensing performance towards ethanol of a Nb_2O_5 thin film intended to be employed for the realization of conductometric gas sensors. The sensing film, deposited by reactive plasma sputtering on a tiny commercial substrate, was characterized in terms of morphology and chemical composition.

Subsequently, sensitivity was assessed at different working temperatures between $250\ ^{\circ}\mathrm{C}$ and $400\ ^{\circ}\mathrm{C}$ and a temperature of $350\ ^{\circ}\mathrm{C}$ resulted as the best tradeoff between sensitivity and power consumption. Sensor sensitivity is quite good allowing one to detect ethanol at the ppm level. Stability and repeatability of the sensor were tested as well, revealing no significant modification of the sensing performance also after repeated measurements and expositions to different gases. Moreover, the sensor revealed almost no saturation up to a concentration of $400\ \mathrm{ppm}$. Such a wide detection range makes the proposed sensor a valid solution for monitoring ethanol in several different applications ranging from chemical and food industry to health care and breath analysis. In particular, the detection range going from few ppm to hundreds ppm make such sensor suitable for the detection of ethanol in the exhaled breath in drunk-driving tests. All tests were performed in a flow-through environment which is not expected to significantly modify the sensor performance in respect to a static environment. Anyway, the possible small variations which can occur in static-environment applications can be compensated by sensor calibration carried out under static conditions.

The only drawback highlighted during the tests was a high sensitivity towards acetone which can be an interferent in some application. In fact, acetone is normally present in exhaled breath, especially in subjects affected by diabetes. A possible

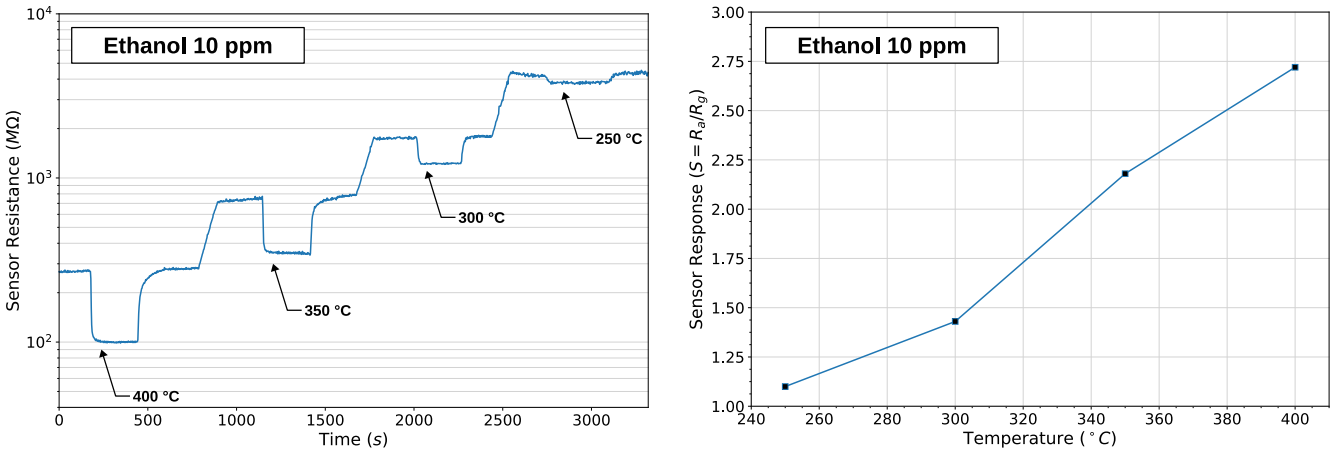

Fig. 4. Sensor response towards 10 ppm of ethanol at different temperatures between 250 °C and 400 °C.

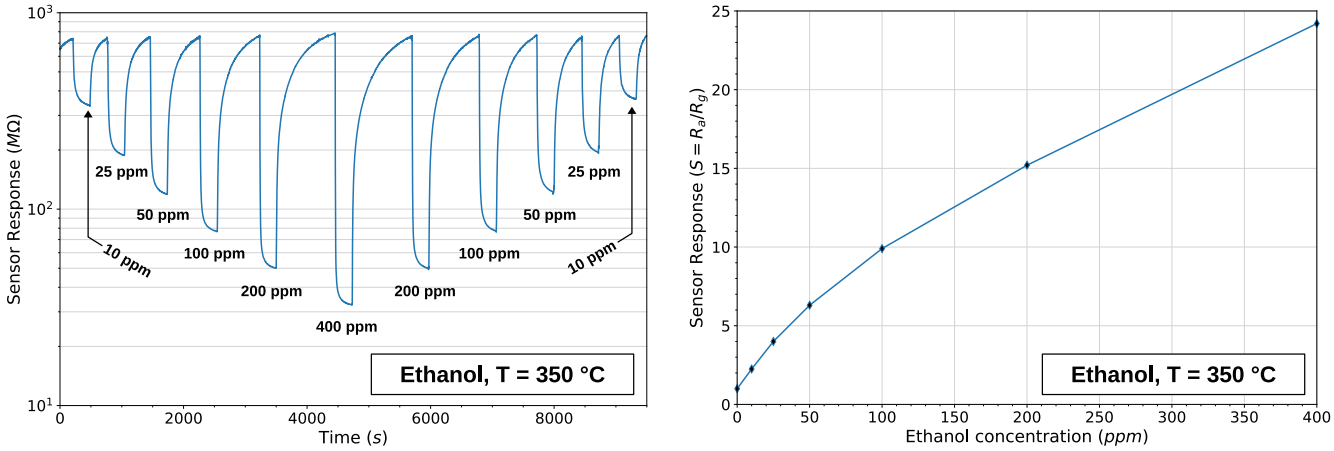

Fig. 5. Relative response of the sensor at a working temperature of 350 °C towards ethanol at several different concentrations between 10 ppm and 400 ppm.

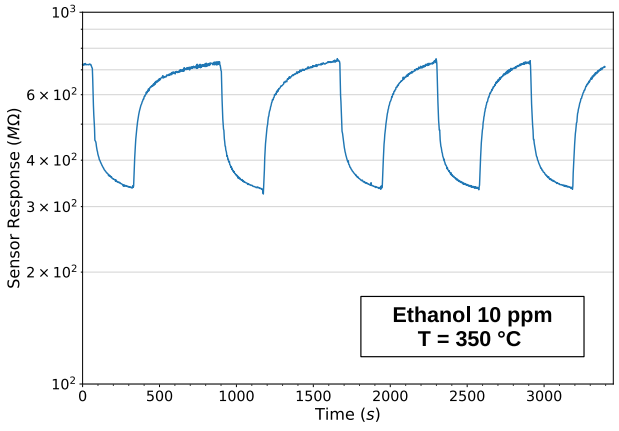

Fig. 6. Sensor response to five repeated ethanol impulses at 10 ppm.

solution to such a problem might be the addiction of catalytic metals, such as gold, silver and platinum, to the niobium oxide in order to improve selectivity. Nevertheless, the achieved results are promising and the proposed sensor, as it is, can be employed in several applications where acetone is not expected.

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