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## Ozone detection based on nanostructured $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$ thin films

S. Smiy, M. Bejar, E. Dhahri, T. Fiorido, Marc Bendahan, K. Aguir

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Fiorido; Marc Bendahan; Khalifa Aguir

Abstract: The present paper describes the synthesis of gas sensors based on thin films perovskite-type La<sub>0.8</sub>Pb<sub>0.1</sub>Ca<sub>0.1</sub>Fe<sub>0.8</sub>Co<sub>0.2</sub>O<sub>(3)</sub> over a  $[\text{SiO}]_2/\text{Si}$  substrate with interdigitated Pt-electrodes using the drop coating method. The perovskite starting nanoparticles have been prepared by the sol-gel method and they have an orthorhombic structure with Pnma space group. The prepared thin film has been tested as a sensitive film for ozone detection and shows a promising response. The detection of low concentration of ozone (equal to 50 ppb) makes the prepared thin film a potential sensor for ozone detection.

## Cover letter

**Pr. Moez BEJAR**

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**22/12/2019**

**Dear Editors of the Journal of Alloys and compounds.**

I am pleased to submit our manuscript entitled:

**“Ozone detection based on nanostructured  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  thin films”**

for consideration for publication in the **Journal of Alloys and compounds**.

It has not been published elsewhere and that it has not been submitted simultaneously for publication elsewhere. In this manuscript, we investigated the response of  **$\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$**  compound toward the Ozone oxidizing gas.

Thank you for your consideration,

Yours Sincerely;

**Pr. Moez BEJAR**

## Prime Novelty Statement

The  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sample has been prepared using the drop coating method. The structural and Ozone gas-sensing properties were reported. The gas sensing properties of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  shows that ours sensor can detect low concentrations of Ozone-gas and we deduced that our sensor can be used as promising candidates to detect Ozone-gas added to that our captor is able to detect this dangerous gas at different concentration.

## Ozone detection based on nanostructured

### $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$ thin films

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#### ABSTRACT

The present paper describes the synthesis of gas sensors based on thin films perovskite-type  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  over a  $\text{SiO}_2/\text{Si}$  substrate with interdigitated Pt-electrodes using the drop coating method. The perovskite starting nanoparticles have been prepared by the sol-gel method and they have an orthorhombic structure with *Pnma* space group. The prepared thin film has been tested as a sensitive film for ozone detection and shows a promising response. The detection of low concentration of ozone (equal to 50 ppb) makes the prepared thin film a potential sensor for ozone detection.

**Keywords:** perovskite, drop-coating, thin films, semiconductor, gas sensor, ozone.

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#### I- INTRODUCTION

Recently, the synthesis of advanced materials for many applications has been widely investigated in literature. In particular, the interest for the perovskite materials  $\text{LnMO}_3$  (Ln: lanthanide and M: trivalent cations), destined for innovative technological applications, such as catalysis, magnetocaloric and gas sensing field, have grown steadily [1-4].

The  $\text{LaFeO}_3$  is a potential functional material which is used in many fields such as gas-sensing in the detection of many gases, including  $\text{HCHO}$ ,  $\text{CH}_4\text{O}$  and  $\text{CH}_3\text{COCH}_3$  [5-9]. Also, derivatives of  $\text{LaFeO}_3$  are very used in the detection of dangerous gases. Song et al. have proven that introducing Pb in the A-site to replace Lanthanum (La) increase the response of  $\text{LaFeO}_3$  material [10]. In addition, the introduction of Ca in  $\text{LaFeO}_3$  has caused the increase of sensitivity of  $\text{La}_{1-x}\text{Ca}_x\text{FeO}_3$  to alcohol [11]. In another study, H.

Saoudi et al. [12] have reported that introducing Cobalt (Co) in B-site in  $\text{La}_{0.8}\text{Ca}_{0.1}\text{Pb}_{0.1}\text{Fe}_{1-x}\text{Co}_x\text{O}_3$  perovskite materials will increase the response because the sample with  $x=0.05$  shows the higher sensitivity of  $\text{NH}_3$ -gas compared to  $\text{La}_{0.8}\text{Ca}_{0.1}\text{Pb}_{0.1}\text{FeO}_3$  sample.

The ozone-gas has been emerged in many fields, e.g. textile, food, water treatment, pharmaceutical and chemical industries [3]. Consequently, ozone constitutes one part of the summer toxic smog. However, for a concentration above 120 ppb in the atmosphere, Ozone could cause serious health problems (e.g. burning eyes, lung damage and respiratory irritation) [13, 14]. Moreover, the air quality is classified referring to the Ozone amount in the atmosphere: very good (0 – 50 ppb), good (50 – 100 ppb), poor (100 – 200 ppb) and very poor (> 200 ppb) [3]. For all the above reasons, the monitoring of the Ozone concentration in our atmosphere is becoming a serious issue.

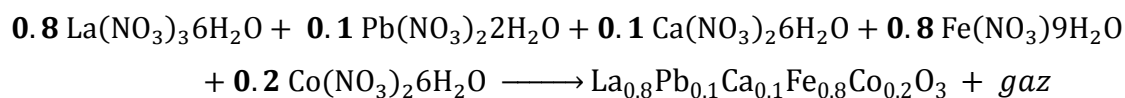
In our previous published work related to this framework, we have demonstrated the high sensing properties of  $\text{La}_{0.885}\text{Pb}_{0.005}\text{Ca}_{0.11}\text{FeO}_{2.95}$  towards the  $\text{NH}_3$ -gas [1].

In line with our interest in the study of gas sensing, we report in the present research the synthesis of a thin film gas sensor based perovskite-type  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  over a  $\text{SiO}_2/\text{Si}$  substrate with interdigitated Pt-electrodes using a drop coating as a new method.

## II- EXPERIMENTAL DETAILS

### II-1) Preparation of perovskite nano-powder

Nano-powder of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  has been prepared by mixing different nitrates:  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{Pb}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{Ca}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  as precursors according to the proportion indicated in the equation below:



Then, the collected powder has been sintered at 300, 600 and 900 °C for 24h for each temperature.

### II-2) Thin film preparation

The perovskite thin-film was deposited over the  $\text{SiO}_2/\text{Si}$  substrate (4 mm x 4 mm) with interdigitated Pt- electrodes by the drop coating method. Firstly, a slurry of 20 mg of

perovskite mixed with 20 ml of 2-propanol has been prepared and ultrasonicated for 10 min at 40 °C to ensure a good homogenization for the mixture. Subsequently, the deposition of the thin film was assured by coating the substrate drop by drop using a micropipette. The process has been combined with heating at 50 °C after each drop to remove the solvent. The thin film properties were optimized at 8 drops which makes a total of 20  $\mu$ l. Finally, after the deposit of 8 drops a heating for one hour at 550 °C will take place (Fig. 1).

### II-3) Ozone sensing measurement:

To investigate the ozone sensing properties of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  films, the sensors have been introduced in a test chamber allowing the control of the sensor temperature under variable gas concentrations. Dry air has been used as a reference gas. Ozone gas was generated by oxidizing oxygen using a pen-ray UV lamp (Stable Ozone Generator UVP/185 nm). The intensity of the UV radiation was varied by shifting a shutter around the lamp. The different ozone concentrations are obtained in the range of 30–800 ppb with a flow rate of dry air maintained at 0.5 l/min.

The operating temperature of the sensor has been adjusted between 160 °C and 280 °C to determine the best sensor response. In this case, our sensor was exposed to 100 ppm ozone-concentration for 1 min in order to identify the best operating temperature.

The applied dc voltage for sensor measurement is 1V and the current is measured using a computerized sourcemeter type Keithley 4250. The sensor response is defined as  $S = R_a/R_g$ , where  $R_a$  and  $R_g$  are the resistance under air and the resistance when the sensor is exposed to ozone-gas, respectively.

## III- RESULTS AND DISCUSSION

### III-1) Scaling analysis

The Rietveld refinement of the X-ray diffractogram of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  nanopowder (Fig. 2) has revealed that our sample crystallizes in the orthorhombic structure with the  $Pnma$  space group. The lattice refined parameters  $a, b$  and  $c$  are 5.5493, 7.8489 and 5.5394 Å, respectively. The  $(hkl)$  Miller indices corresponding to the main reflexion planes of our sensor are (1 0 1), (0 0 2), (2 2 0), (0 4 0), (1 0 3), (3 2 1), (2 4 2) and (3 2 3). Moreover, the analysis using “X0 Pert High Score Plus” program has shown the presence of  $\text{Fe}_3\text{O}_4$  as secondary phase.

The crystallite size ( $D_{W-H}$ ) of the powder has been evaluated using the Williamson-Hall method [15] ( $\beta \cos(\theta) = f(4\sin(\theta))$ ) (Fig. 3). The sensor has shown a nanometric crystallite size equal to 43.870 nm.

$$\beta \cos(\theta) = k\lambda/D_{WH} + 4\varepsilon \sin(\theta) \quad (\text{Eq. 2})$$

Where  $\beta$  is the width at half height,  $\varepsilon$  is the coefficient due to structural defects,  $k$  represents the grain form factor ( $k = 0.9$ ),  $\lambda$  is the radiation wavelength ( $\lambda = 1.5406 \text{ \AA}$ ) and  $\theta$  is the angle corresponding to each peak.

### III-2) Ozone sensing properties:

Fig. 4 shows the variation of the electrical resistance as a function of time under dry air with a temperature increasing from 25 to 180 °C. The results show that the resistance decreases with increasing the temperature, which proves the semiconductor behavior of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor [16].

In order to determine the operating temperature of our compound, the sensor responses in presence of 100 ppb of ozone have been carried out at different temperatures between 160°C and 280°C. Fig. 5 shows the resistance variation versus time at 170, 190, 260 and 280 °C during ozone exposing time of 1 min.

The results show that the sample displays reversibility and the curves present good stability of the baseline. Furthermore, the sample resistance has declined upon exposure to an oxidizing gas [17], indicating the *p*-type semiconductor behavior of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor [18]. In fact, with an oxidizing gas, such as Ozone, the conductivity increases due to the enhancement of the holes of concentration.

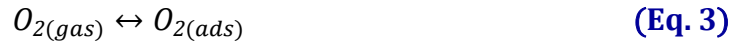
Table.1 summaries the ozone ( $\text{O}_3$ )-responses values of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor. The maximum of the sensitivity ( $R_a/R_g$ ) has been reached at 170 °C when our sensor was exposed to 100 ppb for 1min, 170 °C is considered as the operating temperature ( $T_{Op}$ ) of the sensor.

The sensor performances have also been evaluated with different ozone concentrations and the results are presented in Fig. 6(a). The results exhibit the sensing ability of the thin-film in all the studied ozone concentrations. In the same way, Fig. 6(b) shows that the response intensities increase together with the ozone concentration and reach a maximum of 3.6 for the concentration of 400 ppb. The recorded values are depicted in Table 2. These values are better compared with those reported earlier [19-22].

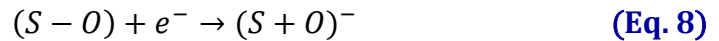


The response time ( $\tau_{resp}$ ) is another important parameter to evaluate the sensor, which is the time required to reach 90 % of the electrical resistance when the sensor is exposed to Ozone gas [1]. The response time values ( $\tau_{resp}$ ), recorded and gathered in Table 3, have revealed that  $\tau_{resp}$  decreases when increasing the concentration at the operating temperature of 170 °C. The same trend was observed previously by Masami Mori and *al.* [3]. In contrast,  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor has low response time values compared to other perovskite materials, such as  $\text{SmFeO}_3$  [3].

The  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  response, signal and response time are influenced by the quantity and quality of oxygen adsorbed on the surface of our sensor [23]. On the surface, atmospheric oxygen can be adsorbed very easily and atoms or molecules become chemisorbed ions  $\text{O}^-$  or  $\text{O}_2^-$ . When the temperature increases the state of oxygen on the surface undergoes the following reactions (from Eq. 3 to Eq. 6) and this phenomenon is demonstrated theoretically by using the  $\text{SnO}_2$  model [24]. In fact, our thin film presents an increase in hole concentration because when oxygen species are adsorbed in  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  the sensor surface will capture electrons from our sensor which is a *p*-type semiconductor and consequently a depletion region of electrons is thus formed [25]. Added to that, at the operating temperature ( $T_{op}$ ) we have a maximum of oxygen adsorbed on the surface of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor [1].



The chemical reactions process between free adsorption site *S* and the triatomic gases, as Ozone ( $\text{O}_3$ ), are [26]:



#### IV- CONCLUSION

In this research paper, the  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor has been successfully prepared by the drop coating method and tested as an ozone sensor. The prepared thin-film showed sensitivity to *ppb*-levels of ozone gas. The resistance study as a function of

time at different working temperatures depicted the good sensitivity of our sensor especially at the operating temperature of  $170\text{ }^{\circ}\text{C}$ . In addition, our sensor can be used in the distinction of the air quality for the ozone and sub-ppm levels detection becomes possible.

Our study shows the possibility of the use of our sensor in the field of the detection of ozone-gas. Another necessary part of our work is the study of the selectivity of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor.

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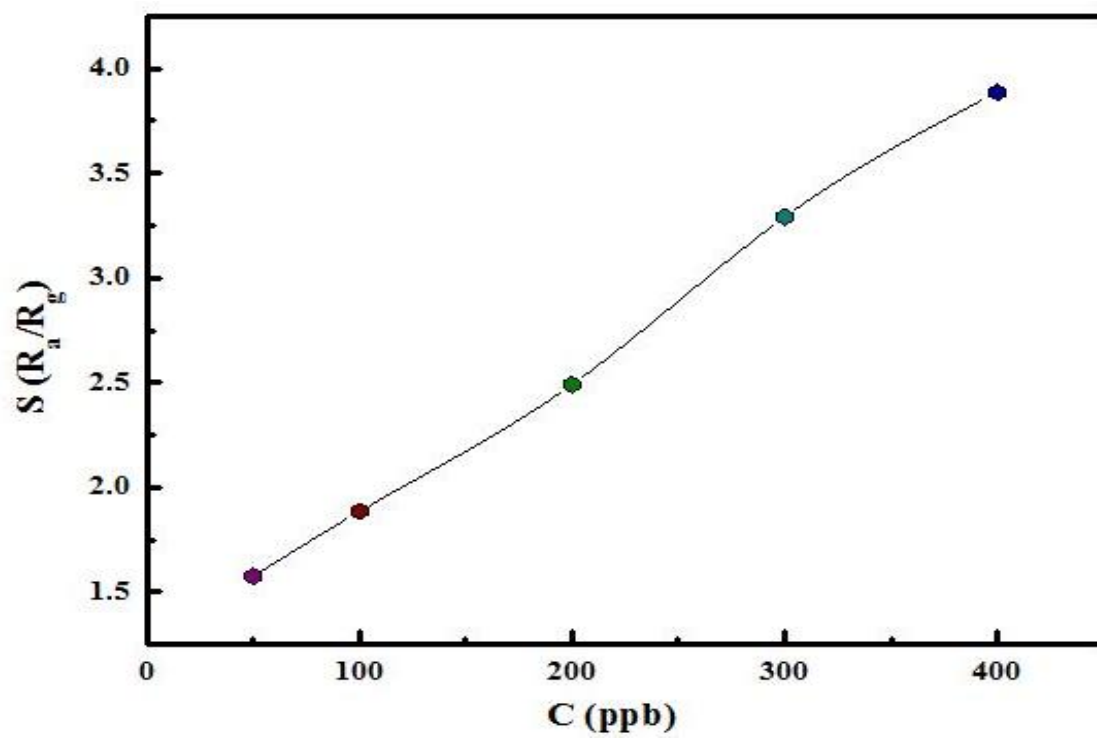
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## Graphical abstract



## Research Highlights

- $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sample crystallizes in the orthorhombic structure with Pnma space group.
- Our compound presents a nano-metric size.
- $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  shows a *p*-type semiconductor behavior.
- $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor can detect the Ozone-gas even with low concentration equal to 50 ppb.
- $T=170^\circ\text{C}$  is the operating temperature of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor.

**Table 1:** The relationship between response ( $S = R_a/R_g$ ) and temperature ( $T$ ) of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  based sensors to Ozone gas.

$T$ (°C)	$S$	$T$ (°C)	$S$
160	1.282	230	1.146
170	1.286	240	1.129
180	1.139	250	1.131
190	1.206	260	1.118
200	1.187	270	1.106
210	1.152	280	1.132
220	1.186		

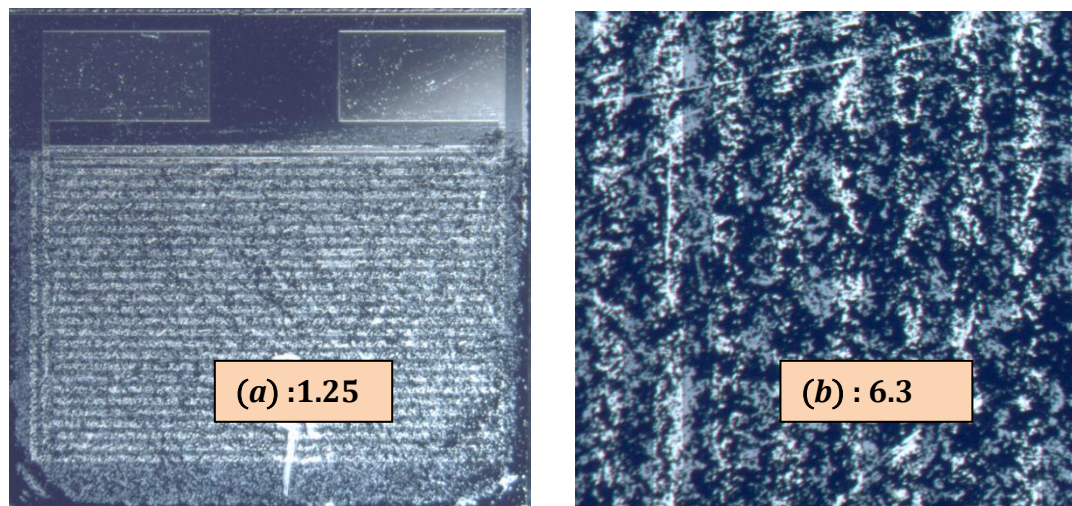


**Table 2:** Response values ( $S = R_a/R_g$ ) of  $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$  sensor (our study) compared to others materials considered for Ozone sensing application.

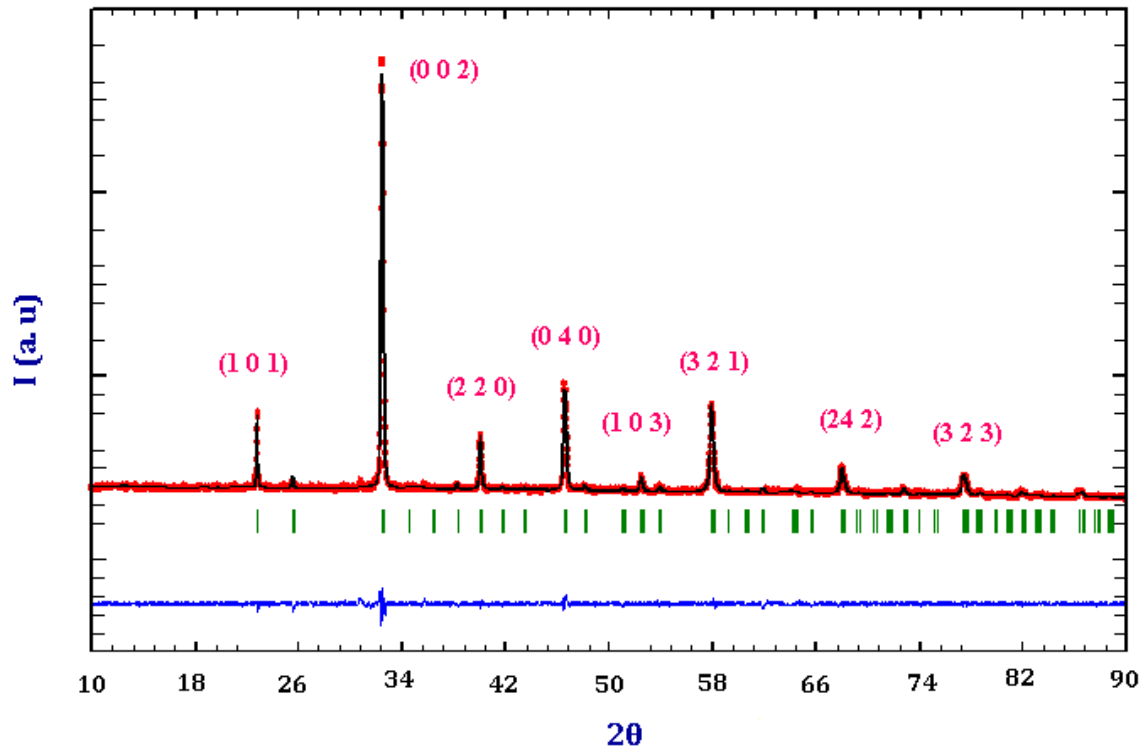
<i>Sensors</i>	<b>T</b> (°C)	<b>C</b> (ppb)	<b>S</b>	<b>References</b>
$WO_3$ (EG : 50 $\mu$ m)	250	200	1.7	[19]
$WO_3$ (EG : 50 $\mu$ m)	250	400	2.3	[19]
$WO_3$ (EG : 50 $\mu$ m)	250	800	2.8	[19]
$WO_3$	300	50	1.25	[20]
$WO_3$	300	100	1.42	[20]
$WO_3$	300	150	1.78	[20]
$WO_3$	300	175	2.25	[20]
$SrTi_{0.925}Fe_{0.075}O_3$	250	75	3	[21]
$SmFe_{0.8}Co_{0.2}O_3$	200	400	3	[22]
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	50	1.6	Our study
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	100	1.9	Our study
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	200	2.5	Our study
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	300	3.3	Our study
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	400	3.9	Our study

**Table 3:** Response times ( $\tau_{resp}$ ) of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor at different concentrations (C) at the operating temperature of 170 °C compared to the  $\text{SmFeO}_3$  sensor.

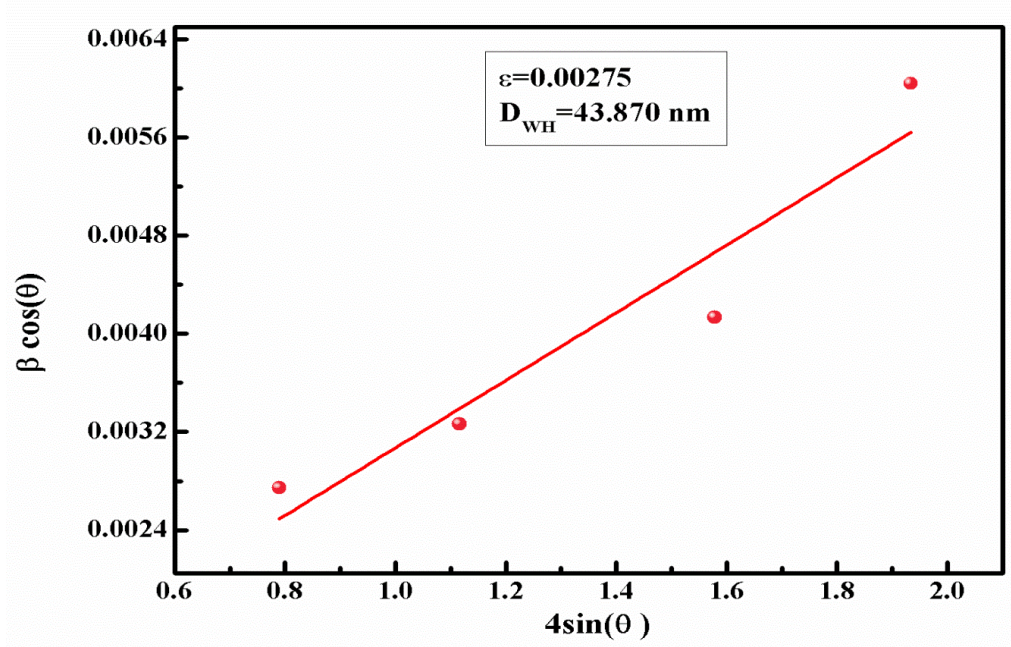
<i>Sensors</i>	<b>T</b> (°C)	<b>C</b> (ppb)	$\tau_{resp}$ (min)	<b>References</b>
<b><i>La<sub>0.8</sub>Pb<sub>0.1</sub>Ca<sub>0.1</sub>Fe<sub>0.8</sub>Co<sub>0.2</sub>O<sub>3</sub></i></b>	<b>170</b>	<b>50</b>	<b>3.5</b>	<b>Our study</b>
<b><i>La<sub>0.8</sub>Pb<sub>0.1</sub>Ca<sub>0.1</sub>Fe<sub>0.8</sub>Co<sub>0.2</sub>O<sub>3</sub></i></b>	<b>170</b>	<b>100</b>	<b>3.0</b>	<b>Our study</b>
<b><i>La<sub>0.8</sub>Pb<sub>0.1</sub>Ca<sub>0.1</sub>Fe<sub>0.8</sub>Co<sub>0.2</sub>O<sub>3</sub></i></b>	<b>170</b>	<b>200</b>	<b>2.5</b>	<b>Our study</b>
<b><i>La<sub>0.8</sub>Pb<sub>0.1</sub>Ca<sub>0.1</sub>Fe<sub>0.8</sub>Co<sub>0.2</sub>O<sub>3</sub></i></b>	<b>170</b>	<b>300</b>	<b>2.2</b>	<b>Our study</b>
<b><i>La<sub>0.8</sub>Pb<sub>0.1</sub>Ca<sub>0.1</sub>Fe<sub>0.8</sub>Co<sub>0.2</sub>O<sub>3</sub></i></b>	<b>170</b>	<b>400</b>	<b>2.1</b>	<b>Our study</b>
<b><i>SmFeO<sub>3</sub></i></b>	<b>200</b>	<b>50</b>	<b>8.4</b>	<b>[3]</b>
<b><i>SmFeO<sub>3</sub></i></b>	<b>200</b>	<b>300</b>	<b>5.2</b>	<b>[3]</b>
<b><i>SmFeO<sub>3</sub></i></b>	<b>200</b>	<b>500</b>	<b>4.0</b>	<b>[3]</b>
<b><i>SmFeO<sub>3</sub></i></b>	<b>200</b>	<b>3000</b>	<b>1.2</b>	<b>[3]</b>



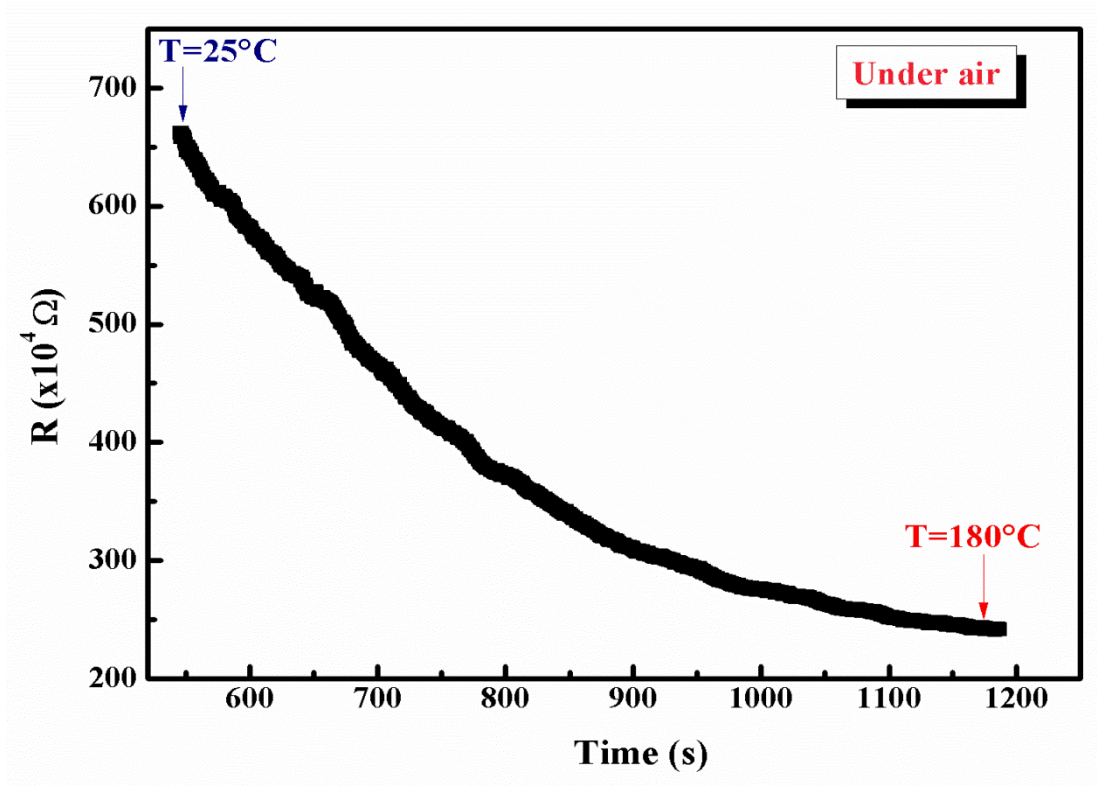
**Figure 1:** (a) Optical sensor view after drop coating of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor and (b) the zoom of the figure.



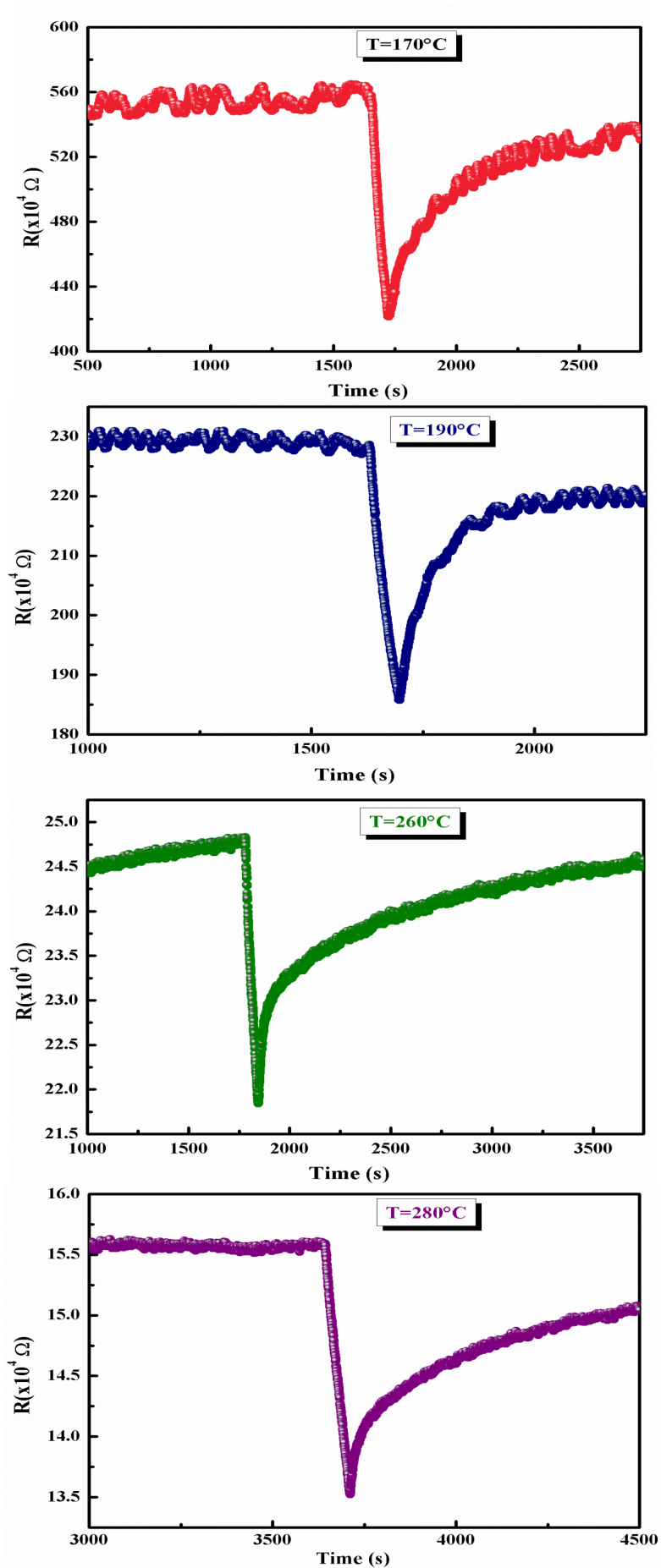
**Figure 2:** Observed (red), calculated (black solid line), their difference patterns (blue) and the Bragg positions (green) of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor and the secondary phase which is attributed to the  $\text{Fe}_3\text{O}_4$  impurity.



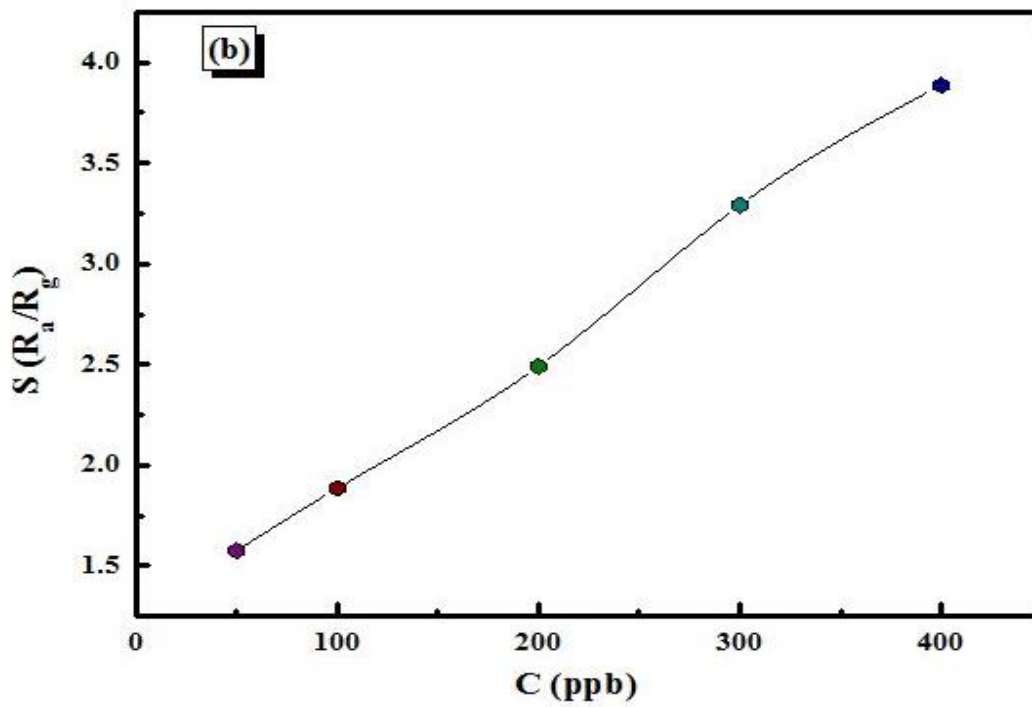
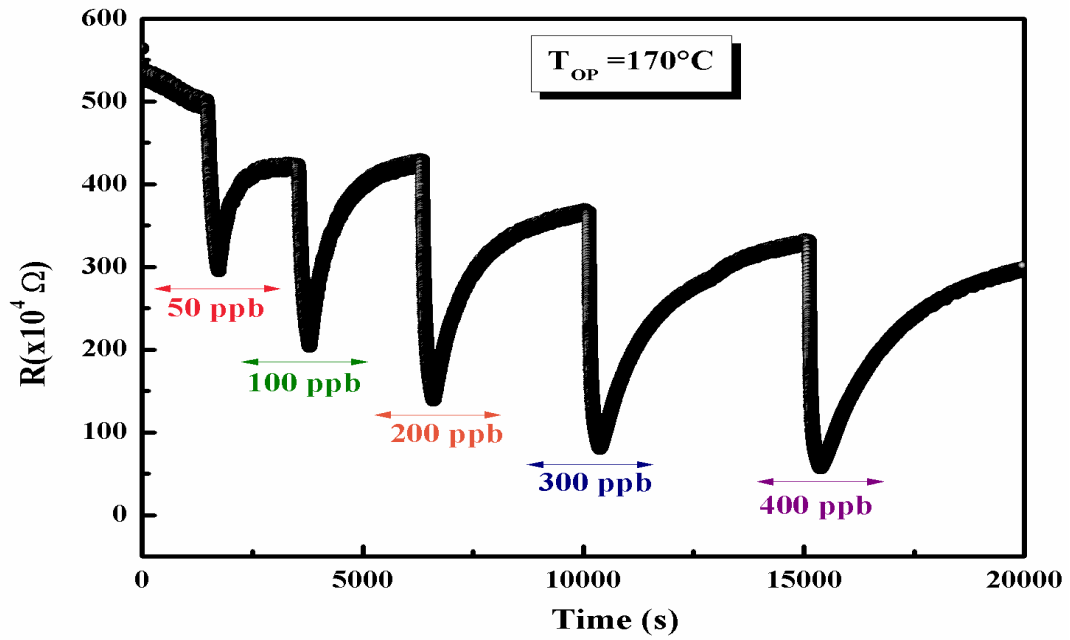
**Figure 3:** The Williamson-Hall analysis plots of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor.



**Figure 4:** Resistance vs. time of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor.



**Figures 5:** Ozone gas response of  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor as a function of time at several temperatures (170, 190, 260 and 280 °C).



**Figures 6:**  $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  sensor response ( $S$ ) to (a) oxidizing gas ( $O_3$ ) and (b) sensor responses depending on the  $O_3$  concentration.



**Declaration of interests**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: