

Ozone detection based on nanostructured L a 0.8 P b 0.1 C a 0.1 F e 0.8 C o 0.2 O 3 thin films

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Abstract: The present paper describes the synthesis of gas sensors based on thin films perovskite-type La_0.8 Pb_0.1 Ca_0.1 Fe_0.8 Co_0.2 O_(3) over a 〖SiO〗_2/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

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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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 **$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$** compound toward the Ozone oxidizing gas.
Thank you for your consideration,

Yours Sincerely;

Pr. Moez BEJAR

Prime Novelty Statement

The $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sample has been prepared using the drop coating method. The structural and Ozone gas-sensing properties were reported. The gas sensing properties of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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.

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Ozone detection based on nanostructured $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ thin films

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ABSTRACT

The present paper describes the synthesis of gas sensors based on thin films perovskite-type $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ over a SiO_2/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 $LnMO_3$ (Ln: lanthanide and M: trivalent cations), destinated for innovative technological applications, such as catalysis, magnetocaloric and gas sensing field, have grown steadily **[1-4]**.

The $LaFeO_3$ is a potential functional material which is used in many fields such as gas-sensing in the detection of many gases, including $HCHO$, CH_4O and CH_3COCH_3 **[5-9]**. Also, derivates of $LaFeO_3$ 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 $LaFeO_3$ material **[10]**. In addition, the introduction of Ca in $LaFeO_3$ has caused the increase of sensitivity of $La_{1-x}Ca_xFeO_3$ to alcohol **[11]**. In another study, H.

Saoudi et *al.* **[12]** have reported that introducing Cobalt (Co) in B-site in $La_{0.8}Ca_{0.1}Pb_{0.1}Fe_{1-x}Co_xO_3$ perovskite materials will increase the response because the sample with x=0.05 shows the higher sensitivity of NH_3-gas compared to $La_{0.8}Ca_{0.1}Pb_{0.1}FeO_3$ 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 $La_{0.885}Pb_{0.005}Ca_{0.11}FeO_{2.95}$ towards the 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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ over a SiO_2/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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ has been prepared by mixing different nitrates: $La(NO_3)_3 6H_2O$, $Pb(NO_3)_2 2H_2O$, $Ca(NO_3)_2 6H_2O$, $Fe(NO_3)_3 9H_2O$ and $Co(NO_3)_2\ 6H_2O$ as precursors according to the proportion indicated in the equation below:

$$\mathbf{0.8}\ La(NO_3)_3 6H_2O + \mathbf{0.1}\ Pb(NO_3)_2 2H_2O + \mathbf{0.1}\ Ca(NO_3)_2 6H_2O + \mathbf{0.8}\ Fe(NO_3) 9H_2O + \mathbf{0.2}\ Co(NO_3)_2 6H_2O \longrightarrow La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3 + gaz$$

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 SiO_2/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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 Fe_3O_4 as secondary phase.

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

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

Where β is the width at half height, ε is the coefficient due to structural defects, k represents the grain form factor ($k = 0.9$), λ is the radiation wavelength ($\lambda = 1.5406$ Å) and θ 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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 (O_3)-responses values of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 (τ_{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 (τ_{resp}), recorded and gathered in **Table 3**, have revealed that τ_{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, $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor has low response time values compared to other perovskite materials, such as $SmFeO_3$ **[3]**.

The $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 O^- or 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 SnO_2 model **[24]**. In fact, our thin film presents an increase in hole concentration because when oxygen species are adsorbed in $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor **[1]**.

$$O_{2(gas)} \leftrightarrow O_{2(ads)} \quad \textbf{(Eq. 3)}$$

$$O_{2(ads)} + e^- \leftrightarrow O^-_{2(ads)} \quad \textbf{(Eq. 4)}$$

$$O^-_{2(ads)} + e^- \leftrightarrow 2O^-_{ads} \quad \textbf{(Eq. 5)}$$

$$O^-_{(ads)} + e^- \leftrightarrow O^{2-}_{(ads)} \quad \textbf{(Eq. 6)}$$

The chemical reactions process between free adsorption site S and the triatomic gases, as Ozone (O_3), are **[26]**:

$$S + O_3 \rightarrow (S-O) + O_2 \quad \textbf{(Eq. 7)}$$

$$(S-O) + e^- \rightarrow (S+O)^- \quad \textbf{(Eq. 8)}$$

IV- CONCLUSION

In this research paper, the $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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 $170\ ℃$. 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 $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor.

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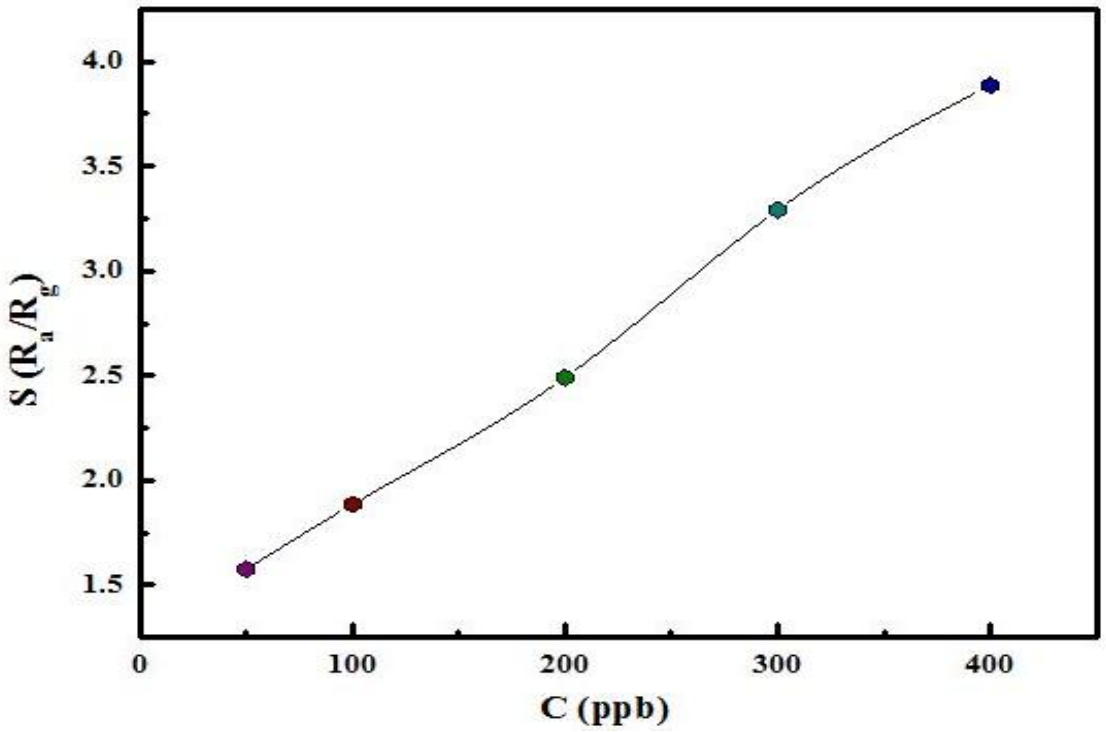
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Graphical abstract



Research Highlights

- $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sample crystallizes in the orthorhombic structure with Pnma space group.
- Our compound presents a nano-metric size.
- $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ shows a *p*-type semiconductor behavior.
- $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor can detect the Ozone-gas even with low concentration equal to 50 ppb.
- T=170°C is the operating temperature of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor.

Table 1: The relationship between response $(S = R_a/R_g)$ and temperature (T) of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ 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.

Sensors	T (°C)	C (ppb)	*S*	References
WO_3 *(EG : 50µm)*	250	200	1.7	[19]
WO_3 *(EG : 50µm)*	250	400	2.3	[19]
WO_3 *(EG : 50µm)*	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 (τ_{resp}) of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor at different concentrations (C) at the operating temperature of 170 °C compared to the $SmFeO_3$ sensor.

Sensors	T (°C)	C (ppb)	τ_{resp} (min)	References
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	50	3.5	Our study
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	100	3.0	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	2.2	Our study
$La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$	170	400	2.1	Our study
$SmFeO_3$	200	50	8.4	[3]
$SmFeO_3$	200	300	5.2	[3]
$SmFeO_3$	200	500	4.0	[3]
$SmFeO_3$	200	3000	1.2	[3]

Figure

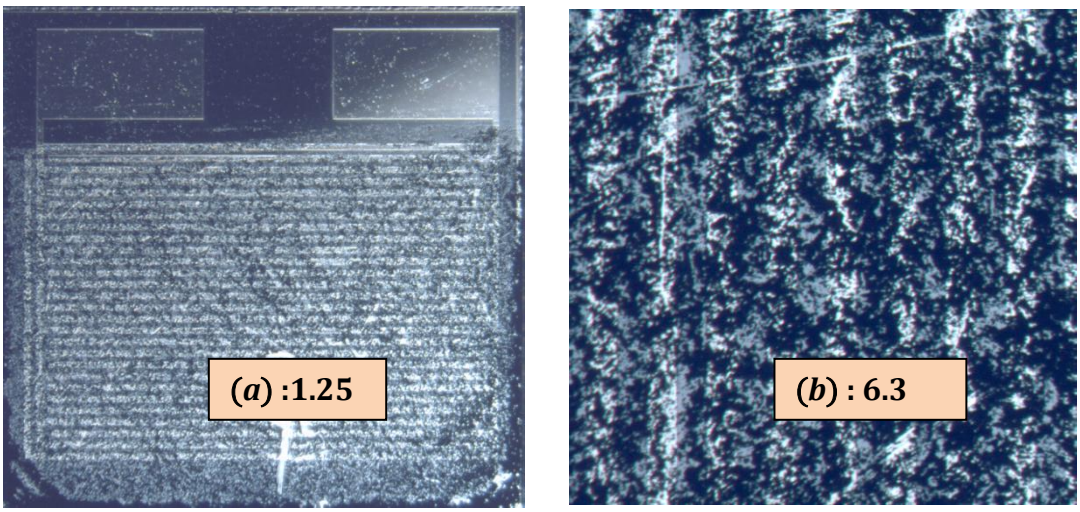


Figure 1: (*a*) Optical sensor view after drop coating of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor and (*b*) the zoom of the figure.

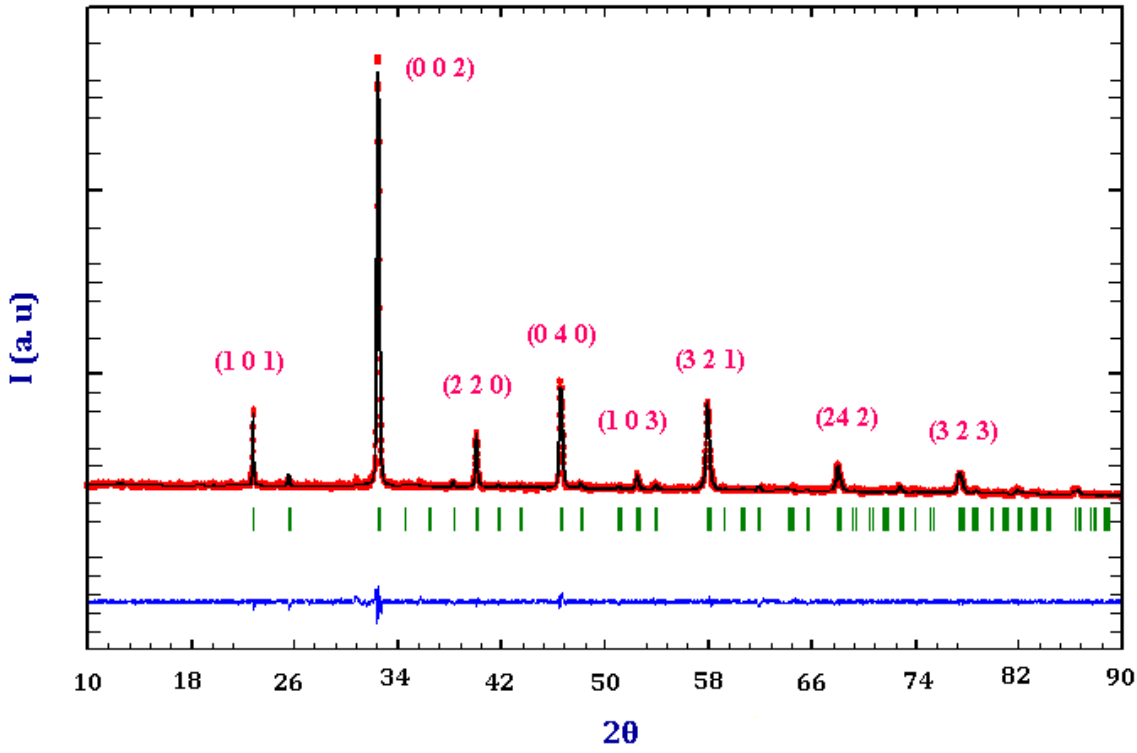


Figure 2: Observed (red), calculated (black solid line), their difference patterns (blue) and the Bragg positions (green) of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor and the secondary phase which is attributed to the Fe_3O_4 impurity.

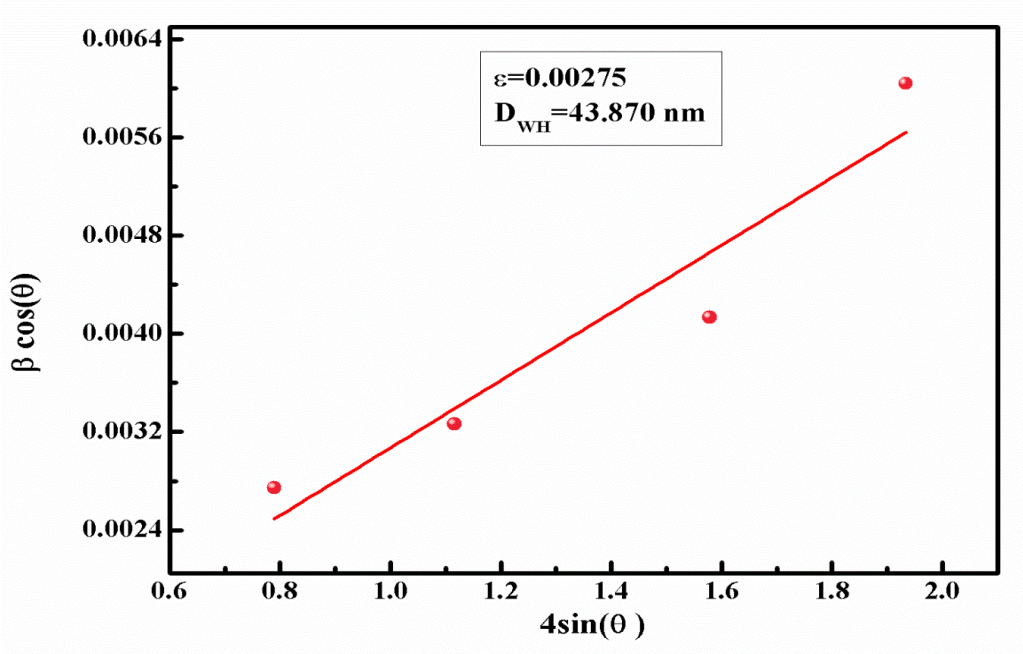


Figure 3: The Williamson–Hall analysis plots of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor.

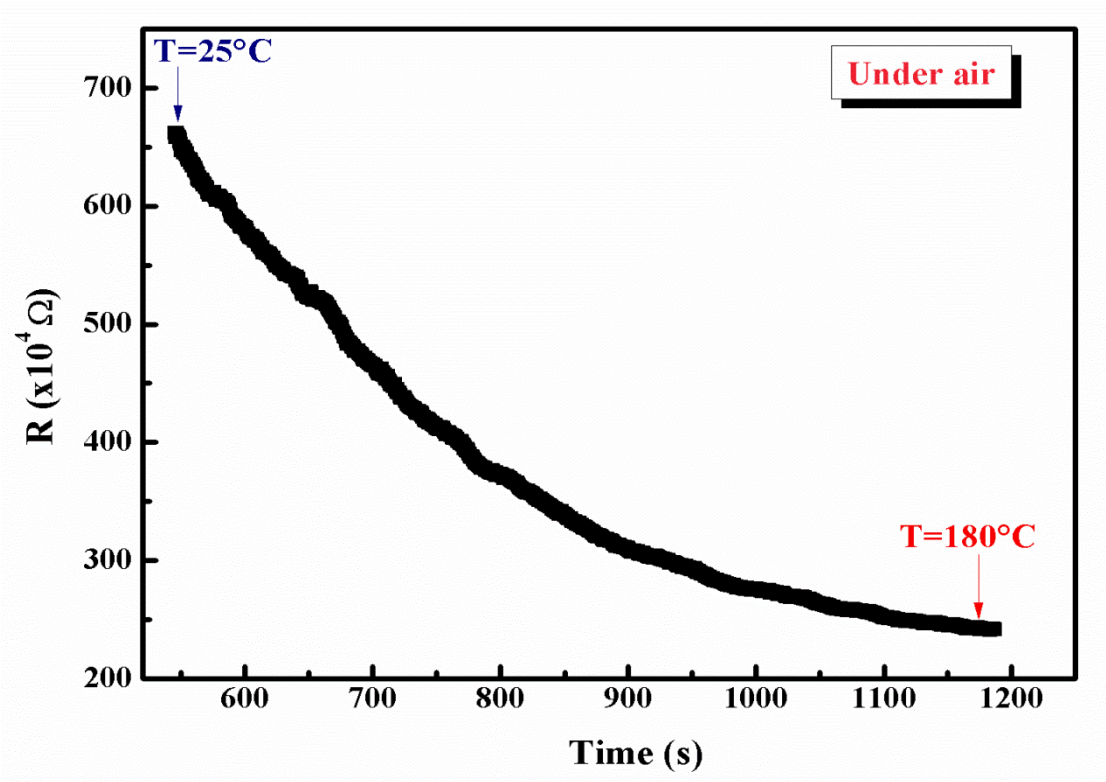
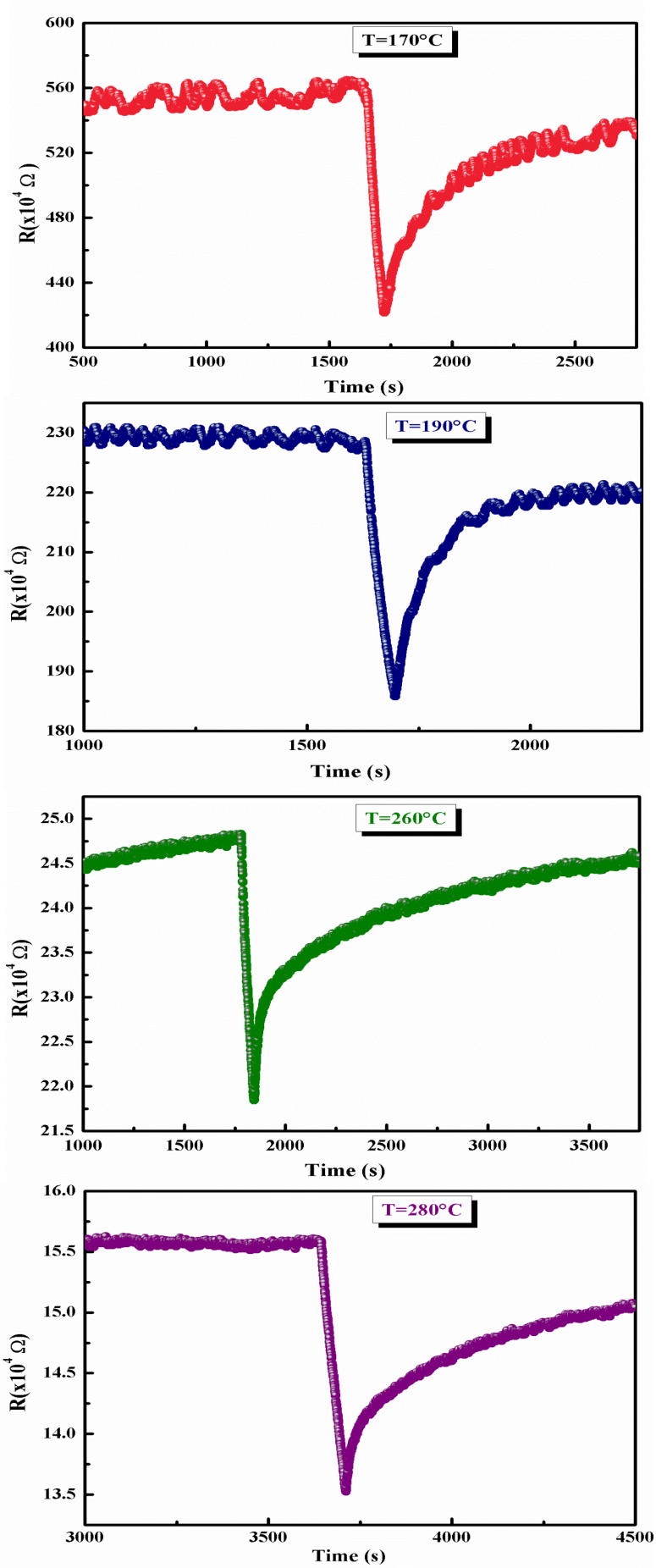
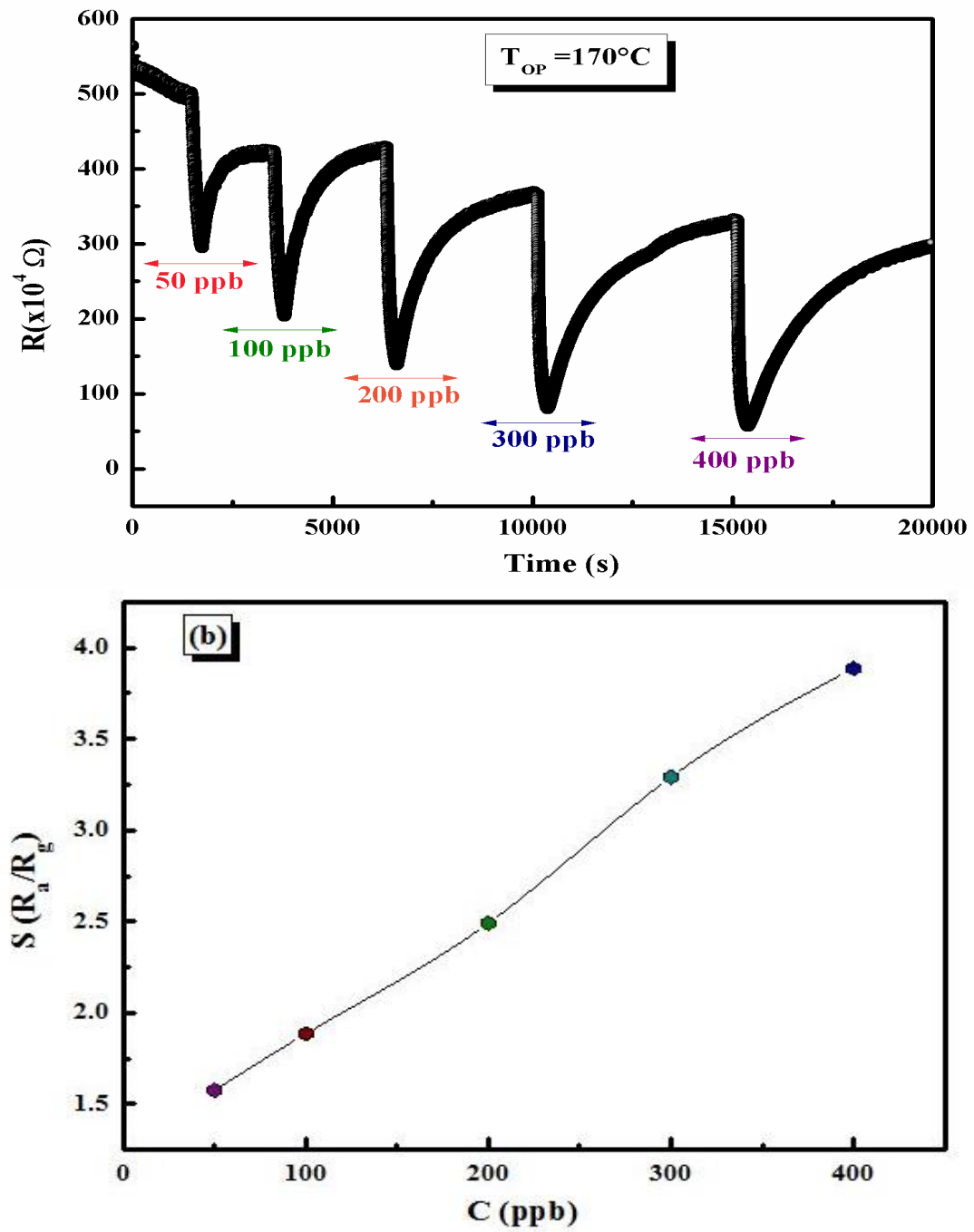


Figure 4: Resistance *vs*. time of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor.



Figures 5: Ozone gas response of $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor as a function of time at several temperatures (170, 190, 260 and 280 °C).



Figures 6: $La_{0.8}Pb_{0.1}Ca_{0.1}Fe_{0.8}Co_{0.2}O_3$ sensor response (S) to ***(a)*** oxidizing gas (O_3) and ***(b)*** sensor responses depending on the O_3 concentration.

***Declaration of Interest Statement**

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: