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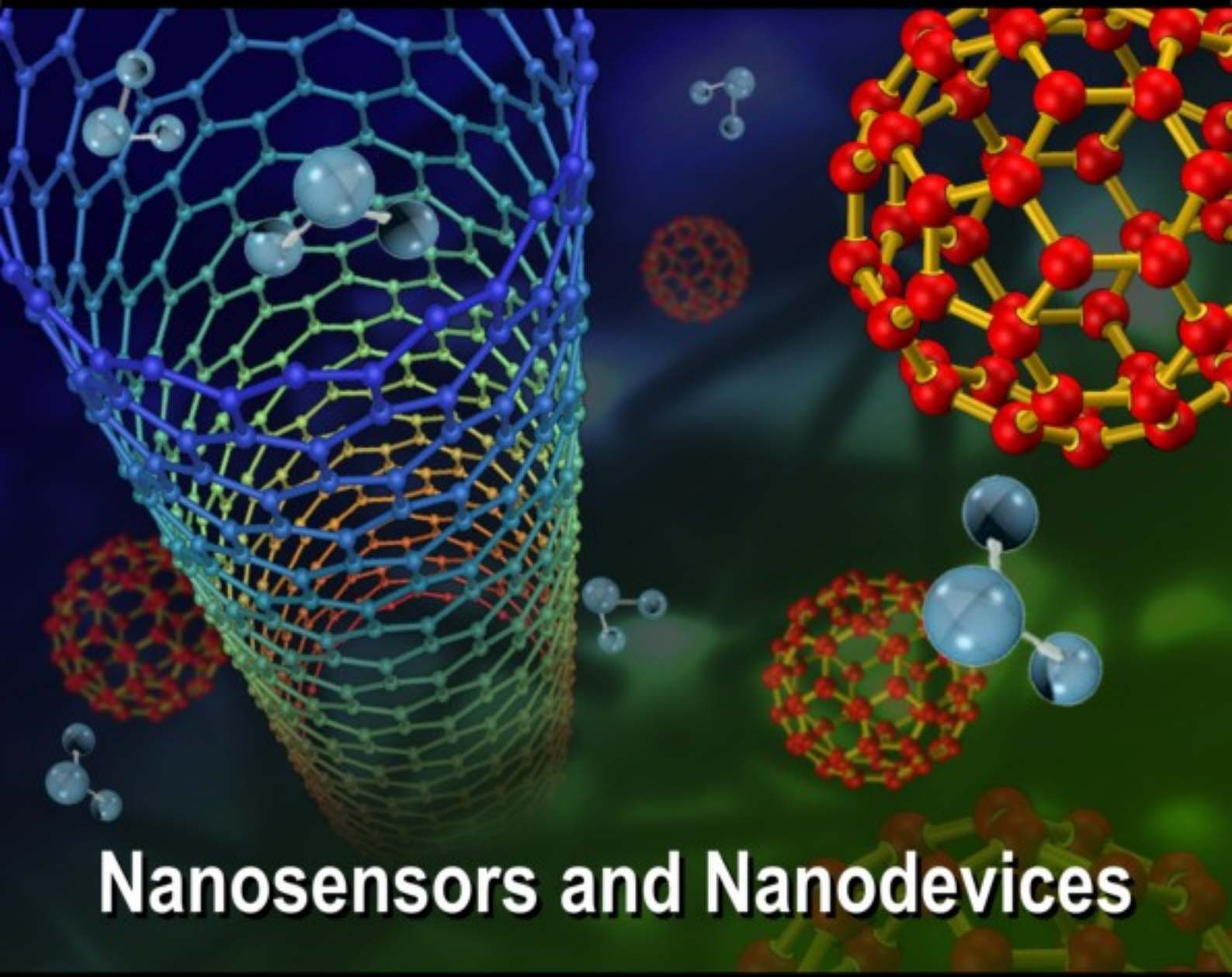

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CO Sensing Properties of Nanostructured $La_{0.8}Sr_{0.2}CoO_3$ Sensors Synthesized by EDTA-Glycol Method

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Abstract: We report a simple method for the preparation of pure $LaCoO_3$ and $La_{1-x}Sr_xCoO_3$ (x = 0.1, 0.2 and 0.25) nanostructures by the EDTA-Glycol method. The final powders obtained by this method have been investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM) measurements. The gas sensitivity of pure and Sr doped $LaCoO_3$ samples were investigated for CO, NH_3, H_2 and LPG. $La_{0.8}Sr_{0.2}CoO_3$ powders (sample GIII) calcined at 650^0C, exhibited a good sensor response towards CO gas at 250^0C. On impregnation of 1 wt.% Pd over sample GIII, the operation temperature reduced to 200^0C with a significant rise in sensitivity. The response time also decreases from about 3.5 min for sample GIII to less than 2.5 min for the Pd loaded element. The electronic interaction between Pd and metal oxide semiconductor is proposed to account for the sensitization effect. *Copyright © 2008 IFSA.*

Keywords: EDTA-Glycol method, Perovskite, Sensitivity, Selectivity

1. Introduction

The development of reliable and selective solid-state gas sensors has great importance both to sense various combustible and toxic gases like CO, H_2, NO_x, SO_x, CH_2, and VOC_S in the field of atmospheric pollution and in the emission control from combustion plants. Recently, many efforts have been aimed to improve the gas sensor performances. Improved selectivity has been obtained by addition of catalysts and/or dopants, while significant improvement of performance can be achieved by decreasing the particle size down to nanometer scale, thus obtaining an increased specific surface area. Semiconductor metal oxides are widely considered as the most promising platform for solid-state gas sensors. Due to enhanced responsiveness of conductance to surface effects in low dimensional

nanostructures, various forms of nanostructured metal oxides have been synthesized and their sensing properties are studied.

ABO_3-type perovskite oxides (A = rare earth element, and B = transition element such as Co, Mn, Ni, Fe, etc) have high potential for their use as catalysts in a number of catalytic reactions [1-6]. The classical ceramic solid-solid reaction and co-precipitation methods, commonly used for the synthesis of perovskite-type oxide, involve high reaction temperature and hence yield perovskite-type oxides with a low surface area due to their sintering [1].

Recently, intensive attention has been paid to $LnMO_3$ perovskite-type oxides due to their applications in solid oxide fuel cells, or materials for technological and chemical sensors [7-10]. $LaCoO_3$ offers much better electrode performance [11, 12] and $LaCoO_3$ based materials exhibit interesting electrical and electrolytic properties. In order to achieve the good performance and functional properties required, dense materials with well-defined microstructures as well as single-phase materials are required. The various methods like, sol-gel [13-15], thermal decomposition [16, 17], combustion of precursors [18] etc. have been adopted for the synthesis of nanostructured $LaCoO_3$. Developments concerning new methods, especially solution techniques involving improvement of the synthesis conditions for obtaining pure phases, have been applied to lower the reaction temperature and to prepare finer and homogenous powders [19-26]. As an alternative to the previously reported methods, we propose the synthesis of pure and Sr doped $LaCoO_3$ by EDTA-Glycol method. To our knowledge, here we report for the first time the obtaining of $LaCoO_3$ based materials by this method. This method has successfully been used for the synthesis of nanostructured $LaCoO_3$ and $La_{1-x}Sr_xCoO_3$ (x = 0, 0.1, 0.2 and 0.25) with homogeneity and very high surface area. The main purpose of this research is to study the sensing properties of nanostructures of $LaCoO_3$ based materials for reducing gases and the effect of Pd loading on sensitivity.

2. Experimental Details

2.1. Synthesis of $La_{1-x}Sr_xCoO_3$ Nanostructures

Nanocrystalline $La_{1-x}Sr_xCoO_3$ powder samples were synthesized for x = 0, 0.1, 0.20 and 0.25 by an EDTA-Glycol method. A calculated quantity of ethylene diaminetetraacaetic acid (EDTA) was firstly dissolved in a small quantity of deionized water followed by the addition of stoichiometric ratio of Co $(NO_3)_2.6H_2O$, La $(NO_3)_3.6H_2O$ and Sr $(NO_3)_2$ with 99.9 % purity. The pH of solution was maintained in the range of 10.3-11.2 in order to facilitate the chelation of metal ions by EDTA. The solution was magnetically stirred on a hot plate at 60^0C for 3 hr in order to get stable metal-EDTA complexes. EDTA is well known ligand with six electron donar centers and having a tendency to form complexes with a large variety of metals. On stirring, appropriate amount of ethylene glycol (EG) was added to this solution. The resultant solution was again stirred for 7 hr on a magnetic stirrer at 95^0C for the removal of excess of water. The prolong heating of the mixture at 95^0C leads into the polyesterification between EDTA-metal complexes and EG which results in the formation of a resin like mass. No turbidity or precipitation was observed during polymerization. The polymeric resin so formed was decomposed by heating at 350^0C, and then subjected for the further heat treatment in a mantle heater at 400-450^0C. This heat treatment fully evaporates highly combustible species from the reaction mixture and burn down most of the organic constituents i.e. induces charring. The resulting ash was slightly ground in to powder and calcined at 650 (samples GI to GIV) and 750^0C (samples SI to SIV) for the period of 6 hr. The obtained powders were then characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. In the present article, results are presented only for X-ray diffraction pattern of samples GI, SI and GIII.

2.2. Impregnation of Pd

Pd was incorporated over the nanostructred sample SIII by the process called; impregnation. The 2 gr. of the sample were added To 100 cm^3 of a $PdCl_2$ aqueous solution of the desired concentration, with HCl ($5 \cdot 10^{-3}$ M). The mixture was vigorously stirred and slowly dried on a water bath. The dried compound was ground to a fine powder and a thermal treatment is applied on some of the obtained powders using a muffle furnace. This treatment consists of 4 hours heating in air at 450 ^{0}C which facilitate the decomposition of palladium chloride.

The different weight percentages of palladium from 0.5 to 1.5 have been used to investigate the noble metal effect on sensing behavior of the samples. Pd impregnated samples are designed as sample GIII:Pd (wt. %).

2.3. Gas Sensing Measurements

Gas sensing properties were investigated at various temperatures from 100 to 300^{0}C. The experiments were performed for all the samples for 1000 ppm CO in air and then for the other reducing gases; NH_3, H_2 and LPG. Conductance measurements were performed in a sealed test chamber with test gases in dry air, at a flow rate of 0.5 l/min. The sensitivity (S), is defined as the ratio:

$$S = \Delta R / R_{\mathrm{AIR}}\,, \tag{1}$$

but

$$\Delta R = R_{\mathrm{AIR}} - R_{\mathrm{GAS}}\,,$$

where, R_{AIR} and R_{GAS} are the resistance in air and in presence of the test gas, respectively.

3. Results and Discussion

3.1. Characterization

The analysis of phase transition has been studied by using X-Rays Diffraction (XRD). With this technique is not only possible to distinguish phase states of the sample, but also to quantify the percentage of each phase in every one of the samples annealing stages. This technique is based on the principle that a monochromatic X-ray, with an incident angle θ respect to a family of crystallographic planes (hkl), is diffracted with an angle 2θ. When this occurs, it is said that there is a reflection and is only produced when Bragg condition is verified (one family of reflections per each family of planes):

$$n\,\lambda = 2\,d_{hkl} \sin\theta\,, \tag{2}$$

where λ is the wavelength of the incident radiation (1.5406 Å for Cu$K\alpha$), d_{hkl} is the distance between the planes (hkl) and n is the refraction order in the family. It can be seen that a correct determination of refraction angle θ conduces to a fast measure of the distance between planes. XRD data of for GI ($LaCoO_3$ calcined at 650^{0}C), SI ($LaCoO_3$ calcined at 750^{0}C), and GIII ($La_{0.8}Sr_{0.2}CoO_3$ calcined at 650^{0}C) shows that the powders are single phase with rhombohedral distorted perovskite structure (Figures 1a and 1b respectively). No secondary phase was observed in these samples. In case of the samples from GI to GIII, it is observed that, the lattice parameters tend to increase with concentration of Sr^{2+} in $LaCoO_3$. The substitution of La^{3+} (r=1.23Å) ions causes an increase of lattice parameters because of the larger ionic radius of Sr^{2+} (r=1.25Å).

Almost complete crystallization of pure and doped $LaCoO_3$ with rhombohedral symmetry takes place at 650^0C. X-ray diffraction pattern of the powder gave some weak unidentified peaks other than $LaCoO_3$ phase. No peaks for the precursor powder were noticed for the EDTA-Glycol method. These observations indicate a better mixing of the cation (Sr^{2+}) in the $LaCoO_3$ perovskite in the formation of sample GIII. The diffraction pattern of the samples GI, SI and GIII are similar and matched well with standard $LaCoO_3$ reflections. Narrow peaks with no significant intensities were seen in the X-ray diffraction patterns of the SI based samples, those are calcined at 750^0C. It is concluded that the calcination temperature leads into grain growth. The broaden peaks suggests that the particle sizes of the samples calcined at 650^0C are small (< 70 nm).

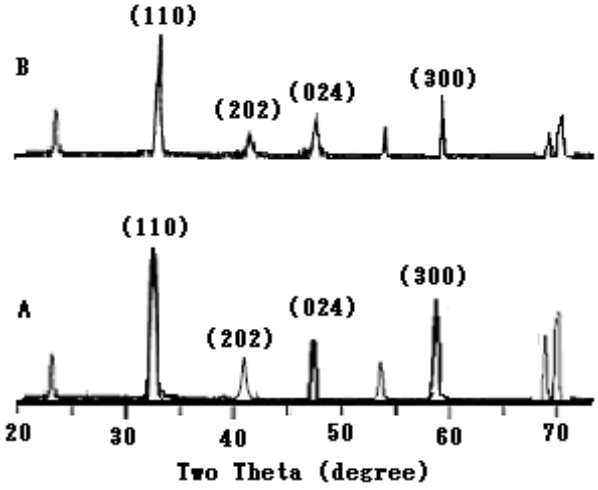

Fig. 1a. XRD pattern of A) $LaCoO_3$ calcined at 650^0C (sample GI) and B) $LaCoO_3$ calcined at 750^OC (sample SI).

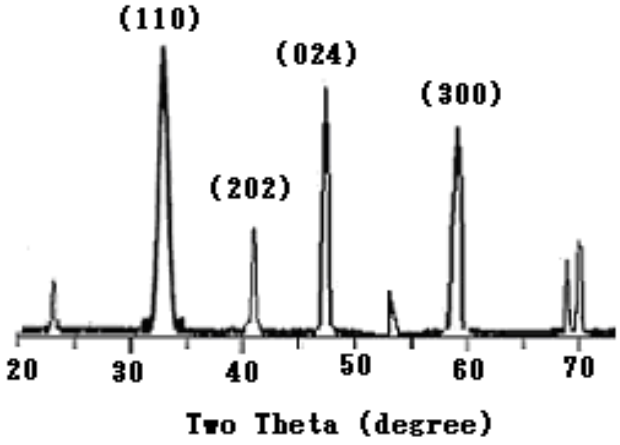

Fig. 1b. XRD pattern of $La_{0.8}Sr_{0.2}CoO_3$ calcined at 650^0C (sample GIII).

3.2. Sensitivity Measurements

3.2.1. Sensitivity of Sample GI and SI

The first tested materials are GI and SI. The sensor responses are represented for CO, NH_3, H_2 and LPG in Fig. 2 and Fig. 3**.** All the responses for both GI and SI are quite low (< 0.5) for all the test gases. The sample GI shows a high sensor response for CO and NH_3 gases at about 300^0C. On the

other hand, the response for H_2 and LPG seems to improve slightly with increasing the operation temperature. No significant improvement in sensing characteristics was seen on increase of calcination temperature in case of SI as shown in Fig. 3. According with XRD results, sample GI calcined at 650^0C has a high quantity lowest grain size. This leads into the provision of a large surface area facilitating the adsorption of oxygen and its reaction with a target gas. However, there are other features, as grain size and porosity, which may also influence the detection. It has been concluded that in the case of the sample SI, these other features are not favorable.

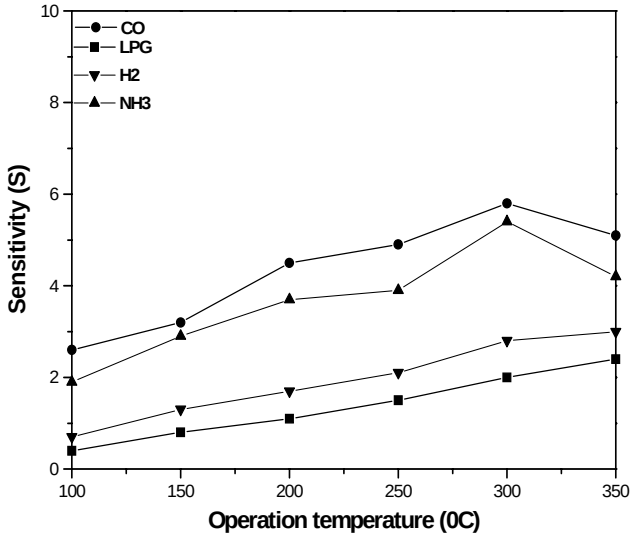

Fig. 2. Sensitivity (*S*) of sample GI towards the reducing gases as a function of an operation temperature (^{0}C).

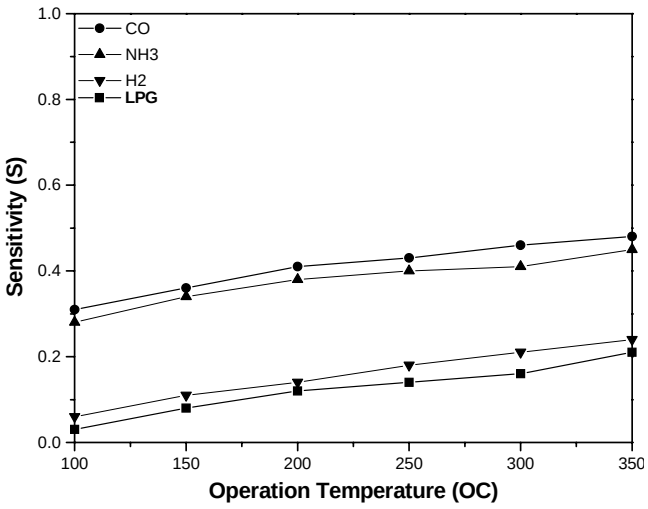

Fig. 3. Sensitivity (S) of sample SI towards reducing gases as a function of an operation temperature (^{0}C).

3.2.2. Sensitivity of $La_{1-x}Sr_xCoO_3$ (x = 0, 0.1, 0.15 and 0.2)

The responses of sensors fabricated with $La_{1-x}Sr_xCoO_3$ based materials toward 1000 ppm of CO gas have been studied at different operation temperatures. A comparison of these responses is shown in Fig. 4**.** An improvement of the sensors responses can be seen for all materials as compared to GI and SI sensors. As it is shown in the XRD results, the addition of strontium ions to the $LaCoO_3$ lattice modifies the physical characteristics of the materials allowing a better interaction with the gas in the

detection process. Moreover, Sr ions also create defects in the material that help the oxygen ions from the gas to be adsorbed [27, 28, 29].

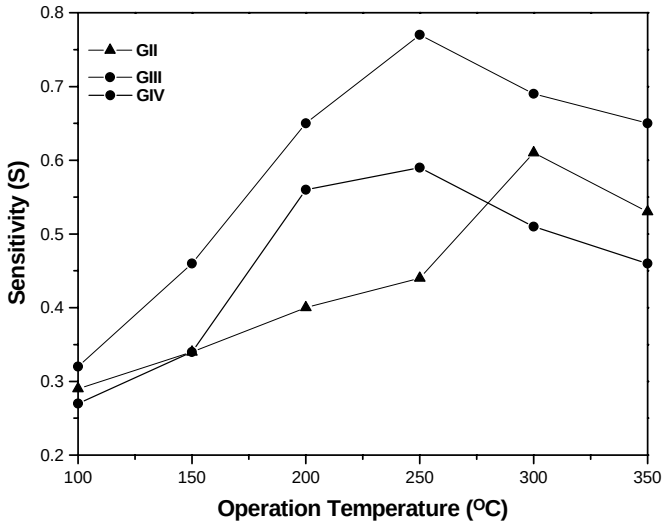

Fig. 4. Sensitivity (*S*) of $La_{1-x}Sr_xCoO_3$ samples calcined at 650^0C for 1000 ppm CO gas as a function of an operation temperature (^{0}C).

The gas sensitivity is usually dependent on the sensor operating temperature and addition. Fig. 4 shows the sensitivity of $La_{1-x}Sr_xCoO_3$ calcined at 650^0C as a function of an operation temperature for the 1000 ppm CO in air atmosphere. It is evident that the sensitivities vary with the amounts of the Sr doping. In the present investigations, doping Sr was found to be the cause of improved sensitivity of $LaCoO_3$ and its selectivity towards CO. The sensitivity for CO increases from samples GI to GIII with increase of Sr concentration and operation temperature. A very high concentration of Sr i.e. x = 0.25 is not significant as shown by the sensor response of sample GIV. A maximum sensitivity for sample GIII to CO at the operation temperature 250^0C; the undoped sample GI gave a maximum sensitivity to CO at 300^0C. From the plot it is clear that, the sensitivity of the $LaCoO_3$ based sensors increases toward CO with addition of Sr and increase in operation temperature. The sensitivity increases with increase in operation temperature and reaches a maximum value corresponding to an optimum operation temperature; 250^0C.

Following the literature, in formation of $La_{0.8}Sr_{0.2}CoO_3$, strontium ions solubility takes place on a large scale as the Sr^{2+} ions have almost similar size to La^{3+} ions [30]. This replacement of ions increases the amount of oxygen vacancies in the sensor material [31] that leads into enhancement of oxygen adsorption and its sensitivity. Being a p-type semiconductor, the holes are charge carriers in $LaCoO_3$. When La^{3+} ions are replaced by Sr^{2+} ions, the carrier's concentration will depend on the number of holes produced by ionization of [Sr_{La}^{x}]:

$$Sr_{La}^{x} \rightarrow Sr_{La}' + h'$$

In this formula, Sr_{La}^{x} mean the point defect, which produced when Sr^{2+} occupies the sites of La^{3+} in the crystals. On the addition of Sr^{2+}, holes will be generated based on this equation. So the concentration of 'h' increases, which in turn increases the conductivity as well as sensitivity of $LaCoO_3$ sensors. It has been reported that the addition of nickel in tin oxide prevent the grain growth [33-38]. The same can be expected in case of the Sr doping in $LaCoO_3$.

The effect of calcination temperature on the gas sensitivity was studied for $La_{1-x}Sr_xCoO_3$ sensors. The samples SI, SII, SIII and SIV; calcined at 750^0C were tested for CO gas at various operation temperatures and shown in Fig.5. In Fig. 6 it is possible to see that the responses of all the samples are not well identified and the sensitivity lies in the range of 0.15 to 0.5. The highest sensitivity observed for sample SIII at 250^0C is about 0.5. For sample SV, the optimum sensitivity recorded was 0.50 at 250^0C which is much less as compared to that observed for GIII at the same temperature. So, the lower dimension of the crystallite size of GIII makes this material more sensitive.

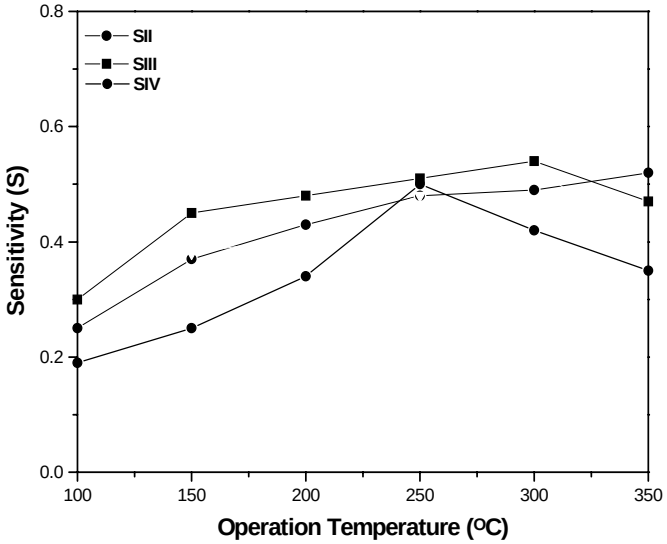

Fig. 5. Sensitivity (*S*) of $La_{1-x}Sr_xCoO_3$ samples calcined at 750^0C for 1000 ppm CO gas as a function of an operation temperature (^{0}C).

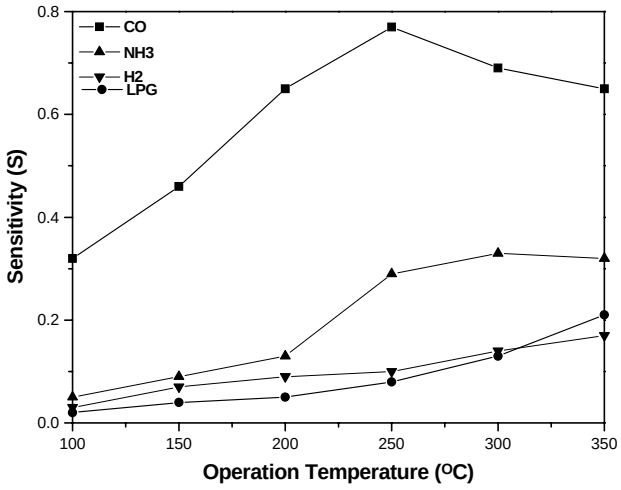

Fig. 6. Sensitivity (*S*) of sample GIII towards CO, NH_3, H_2 and LPG as a function of an operation temperature (^{0}C).

Since sensors based on material GIII ($La_{0.8}Sr_{0.2}CoO_3$ calcined at 650^0C) working at 250^0C have shown the best performance in detecting 1000 ppm CO gas, it has been tested against the other reducing gases like LPG, NH_3 and H_2 as a function of an operation temperature. As can be seen from Fig.6, the sensitivity of this sensor is remarkably higher for CO than for other gases. For these experiments, the

resistance was measured during cooling the sample after being heated to sufficiently high temperature, thereby securing a good reproducibility of the sensitivity-temperature characteristics.

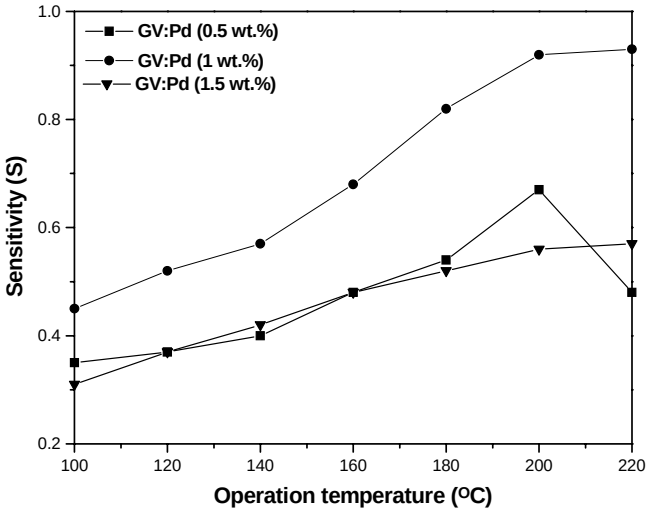

Fig. 7. Sensitivity (*S*) of Pd loaded samples for 1000 CO gas as a function of an operation temperature (^{0}C).

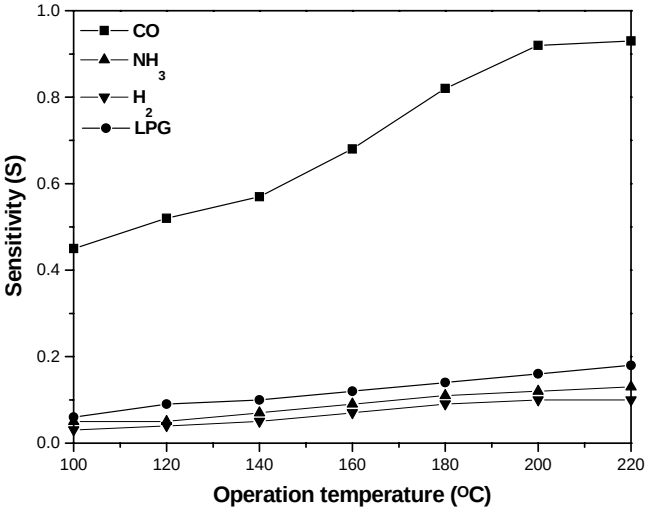

Fig. 8. Sensitivity (*S*) of sample GIII:Pd (1 wt. %) as a function of operation temperature (^{0}C) to CO, NH_3, H_2, and LPG.

3.2.3. Sensitivity of Pd Impregnated Sample GIII

The first tested material was GIII:Pd. The results are represented in Fig. 9. It can be seen from the Fig. 9, that the 1 wt. % Pd is the optimum concentration for the maximum response to CO at 200^{0}C. The response magnitude increased when Pd concentration was increased from 0.5 to 1 wt. % and then decreased above 1 wt. %. It suggests importance of the dispersion of Pd on the semiconductor surface. Indeed the sensitivity is about 96% than that shown by sample GIII. This is consequence of two effects: the initially larger amount adsorbed oxygen and the enhanced reaction of CO with oxygen due to presence of Pd. In other words we can say that, the presence of Pd increases the thick films sensitivity and catalyzes the reaction of CO with previous adsorbed oxygen. The reduction in operation temperature form 250^{0}C to 200^{0}C is considered to be very effective from the commercial point of view.

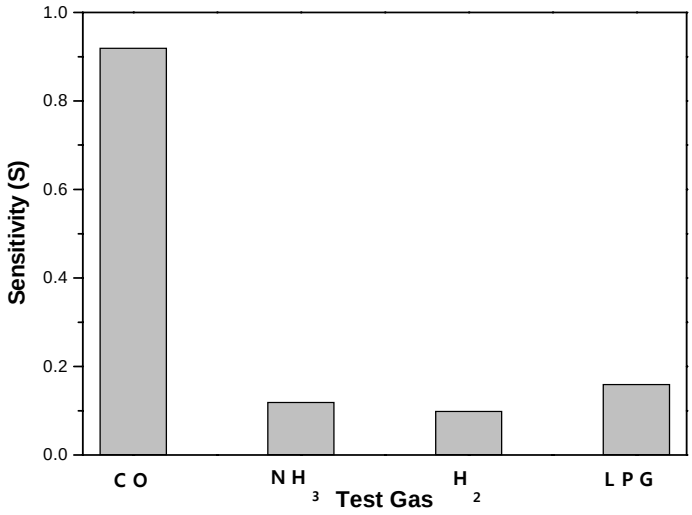

Fig. 9. Sensitivity (*S*) of sample GIII:Pd (1 wt.%) at 200 ^{0}C to various gases.

To check out the cross sensitivity of the sample GIII:Pd (1 wt. %), it was tested for other reducing gases as a function of operation temperature as shown in Fig.9. As per our expectations, the high sensing response can be observed for CO and much less i.e. below 0.2 for the other gases.

Fig. 10 shows the response of 1 wt. % Pd-loaded sample GIII as a function of CO gas concentration at 200^{0}C. It is observed that the response increases with increasing the gas concentration from 100 ppm, but get saturated at higher concentrations above 700 ppm. The sensor element is able to detect CO even up to 100 ppm in air at 200^{0}C with the sensitivity of about 0.23. One of the important parameters for the practical application is the material is the response time for the particular gas. Fig. 11 shows the response curve of the sample GIII:Pd (1 wt.%) to 1000 ppm of CO at 200^{0}C and sample GIII at 250^{0}C. It clearly brings out the fast response and high sensitivity to CO. When compared, the response of sample GIII:Pd (1 wt.%) to CO is fast (< 2.5 min) as than the time taken by sample GIII (> 3 min). As shown in Fig. 11, for sample GIII:Pd (1 wt.%), the 70% recovery can be seen after 6 min, which in turn indicates the excellence in its sensing characteristics.

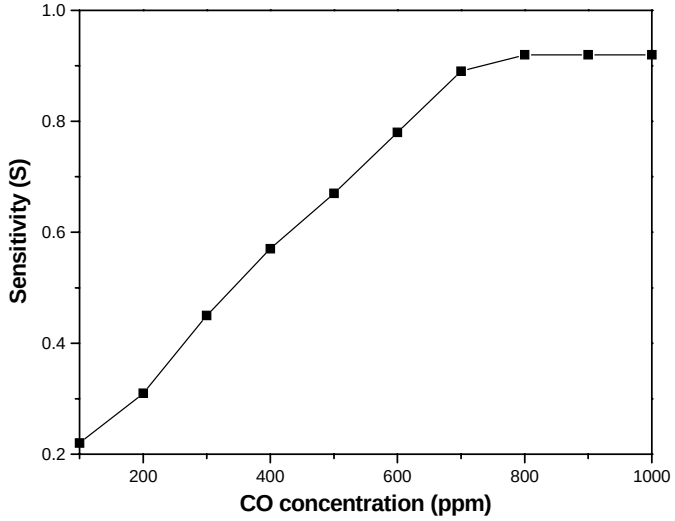

Fig. 10. Sensitivity (*S*) of sample GIII:Pd (1 wt.%) as a function of CO concentration (ppm) at 200^{0}C.

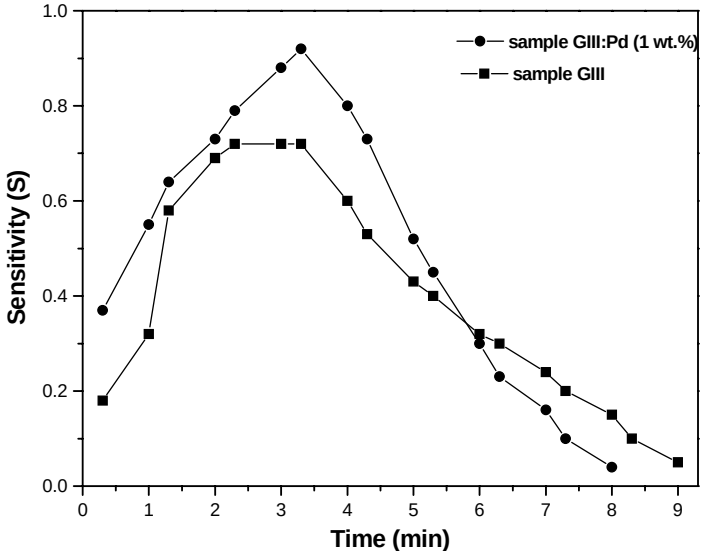

Fig. 11. Response curves for 1000 ppm CO in air for the samples GIII:1 (1 wt.%) at 200^{0}C and GIII at 250^{0}C.

In order to modify or control the surface properties of the sample GIII, introduction of noble metal additives is usually performed. The most important effects of noble metal addition are the increase of the maximum sensitivity and the rate of response, as well as the lowering of the temperature of maximum sensitivity. All these effects arise as a consequence of the promoting catalytic activity when loading with noble metals.

There is an electronic interaction between the added particles and the semiconductor through the space charge created in the semiconductor by the presence of the surface clusters. Additives at the surface of the semiconductor act as receptors while the semiconductor acts as a transducer of the changes taking place at the surface under gas adsorption. This type of sensitization has so far been observed in the metal oxides impregnated with Ag, Pd and Cu, which form stable metal oxides when exposed to air. It was reported in several works, that oxidation of Pd is possible in the range of temperature from 300-450^{0}C, PdO is a predominant species and is responsible for catalytic activity [38]. It has also been observed that the work function of metal oxide changes owing to change in oxidation state of Pd, as it happens in case of Pd/SnO_2 [39]. In other words, Pd in the oxygen phase acts as an electron acceptor and traps the charge from conduction band of metal oxides. This trapped charge, along with that of ionosorbed oxygen, induces a stronger depletion of electrons from the metal oxide surface causing a rise in resistance. Since the depletion of carriers is related to density of trapped charge at the surface, Pd clusters having a size of few nanometers are necessary to affect the resistance.

With Pd on metal oxide, at low temperatures (T<100^{0}C) the rate of CO and oxygen adsorption is high, and the room temperature is enough for the activation of the CO oxidation on the metal additive surface, leading to an electronic exchange between the additive and the metal oxide matrix [40]. The sample response, the magnitude and the growth rate of the conductivity under CO are maximum and the response time is minimum. By increasing the temperature further, the oxidation of CO becomes possible on all the surface of sensor. The area of the working surface increases and, as well as in the case of low temperatures, the saturation value is quickly reached. Owing to the prevalence of desorption processes over adsorption ones, the quantity of adsorbed CO in this temperature range is smaller, leading to a diminution of the conductance. To evaluate the presented relation in accordance with the sensitivity mechanism, the reaction routes need to be studied further.

4. Conclusions

$LaCoO_3$ based powders were successfully synthesized by EDTA-Glycol method. The advantages offered by this method are the low crystallization temperature and the formation of homogenous microstructure of the powder obtained. The sample GIII ($La_{0.8}Sr_{0.2}CoO_3$ calcined at 650^0C) has good sensing properties for CO with high sensitivity at 250^0C. Moreover, its sensitivity and selectivity has been increased for CO with a significant reduction in operation temperature from 250^0C to 200^0C. Sample GIII seems to be promising candidates for monitoring the CO gas at lower temperature 200^0C.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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