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## Ethanol Gas Sensing Properties of Nano-Porous LaFeO<sub>3</sub> Thick Film

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**Abstract.** The characterization and ethanol gas sensing properties of nanoporous LaFeO<sub>3</sub> thick films were investigated. Thick films of LaFeO<sub>3</sub> were prepared by the screen printing technique. Thick films of nanoporous LaFeO<sub>3</sub> were observed to be highly sensitive to ethanol vapours at 300 °C. Upon exposure of ethanol vapours, the barrier height would decrease greatly leading to drastic increase in conductance. It is reported that the surface misfits and operating temperature can affect the microstructure and gas sensing performance of the sensor. The quick response and fast recovery are the main features of this sensor. The effects of microstructure and additive concentration on the gas response, selectivity, response time and recovery time of the sensor in the presence of ethanol vapours were studied and discussed.

**Keywords:** Nanoporous LaFeO<sub>3</sub>, XRD, SEM, TEM, Gas response.

### 1. Introduction

There has been much interest in perovskite-structured compounds (of general formula ABO<sub>3</sub>) because of their unique catalytic action [1], colossal magneto resistance effects [2, 3], and gas-sensing properties [4–12]. The materials are highly defective and have oxygen deficient structures, in which the valence state of metal ions may be controlled by temperature; oxygen partial pressure and dopants [13]. Actuators based on ABO<sub>3</sub>-type oxide materials of rare-earth elements have an excellent stability.

Environmental safety is one of the serious concerns for the scientific community because of rapid increase in the use of organic pollutants in agriculture and various industries which caused severe adverse effects on the environment. Among various organic pollutants, ethanol, a colorless liquid, is one of the pollutants which is widely used as an industrial solvent for the preparation of various materials [14, 15]. It easily mixed with water and hence causes oxidative damage of brain, stomach,

liver, erythrocyte, etc. [16, 17]. Due to its high-toxic nature, it is highly needed to detect efficiently the presence of ethanol in the environment. In this regard, various methods have been developed for the efficient detection of ethanol in environment and are reported in the literature [18–24]. Amongst various detection methods, the electro analytical technique is one of the most important methods for the detection of ethanol as it provides direct, sensitive and reliable analytical results just by utilizing conventional and easily available laboratory electrodes. For the fabrication of efficient electrochemical sensors, mostly artificial mediators are used which helps to transfer the electrons from the electrodes to the detection species. Recently, biocompatible nanomaterials, especially metal oxide nanomaterials, are utilized as artificial mediators for the fabrication of efficient electrochemical sensors [20–24]. LaFeO<sub>3</sub> is considered as one of the promising artificial mediators due to its own properties and functionalities [25, 26]. It is a p-type metal oxide semiconductor with narrow band gap (1.2 eV) and

demonstrated a versatile range of applications. Due to the good electrical conductivity of  $\text{LaFeO}_3$ , it is widely used in the fabrication of various efficient electrical, optical, photovoltaic, sensing and other devices and is reported in the literature [25].

In this work, a successful synthesis of  $\text{LaFeO}_3$  nano sheet via combustion process was done and characterized in terms of their structural and optical properties. Importantly, as-synthesized  $\text{LaFeO}_3$  nanosheets were efficiently utilized as redox mediator for the fabrication of efficient ethanol chemi-sensor which exhibits good sensor performance, specifically in terms of its sensitivity and detection limit.

## 2. Experimental

Analytical grade  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and  $\text{C}_2\text{H}_5\text{NO}_2$  were used as raw materials to prepare  $\text{LaCrO}_3$ . According to the stoichiometric preparation of the reactants, the specified amounts of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  were first dissolved in glycine solution to form the sol. The molar amount of glycine was double to total molar amount of metal nitrates in the solution. A small amount of ammonia aqueous was slowly added to adjust the pH to 7. During this procedure, the solution was continuously stirred and kept at a temperature of 60-70 °C. Then, the stabilized nitrate–glycine sol was poured into a tray and heated slowly to 140-150 °C. Viscosity and color changed as the sol turned into a green, puffy, porous dry gel. When ignited at any point of the dried gel in air at room temperature, the dried gel simultaneously burnt in a self-propagating combustion manner until they were completely transformed into loose powders, known as as-synthesis powder. The entire combustion process would last a few seconds. The as-synthesis powder was the nanocrystalline  $\text{LaFeO}_3$  with fine crystal structure.

### 2.1. Thick Film Preparation

The thixotropic paste was screen printed on a glass substrate in desired patterns [21]. The films prepared were fired at 550 °C for 24 h. These films were surface modified by dipping them into a 0.01 M aqueous solution of cupric chloride for different intervals of time and were dried at 80 °C followed by firing at 550 °C for 24 h in air ambient. The  $\text{CuCl}_2$  dispersed on the films was oxidized in firing process, and sensor elements with different mass% of  $\text{LaCrO}_3$  were obtained. Silver contacts were made by vacuum evaporation for electrical measurements. [13, 25]

### 2.2. Characterization

X-ray diffraction ( $\text{CrK}_\alpha$ , Philips analytical XRD B.V.PW-1710) was used for confirming the phase

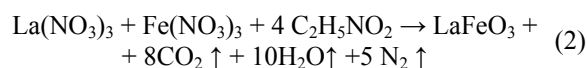
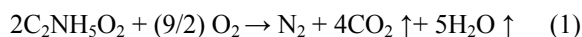
and structural study. Infrared spectra (IR) for the synthesis powder were performed on a Perkin-Elmer 883 spectrophotometer from 450 to 4000  $\text{cm}^{-1}$ . The surface morphological features were observed by using scanning electron microscopy (JSM-6360A). Transmission electron microscopy (TEM) of the powder was carried out by Make Philips CM-200 superwinster spectrometer at 200 kV. The specific surface area of the powders was measured by BET isotherm technique with nitrogen absorption. Thickness measurements were carried out using a Taylor- Hobson (Talystep, UK) system. Electrical and gas sensing characteristics were measured using a static gas sensing system. The I-V character and thermoelectric measurement of nanocrystalline  $\text{LaFeO}_3$  re carried out. The dc resistivity was measured by the two probe method as a function of temperature (room temperature to 550 °C.)

### 2.3 Details of the Gas Sensing System

The sensing performance of the sensors was examined using a static gas sensing system. There were electrical feeds through the base plate. The heater was fixed on the base plate to heat the sample under test up to required operating temperatures. The current passing through the heating element was monitored using a relay with adjustable ON and OFF time intervals. A Cr–Al thermocouple was used to sense the operating temperature of the sensor. The output of the thermocouple was connected to a digital temperature indicator. A gas inlet valve was fitted at one of the ports of the base plate. The required gas concentration inside the static system was achieved by injecting a known volume of test gas using a gas-injecting syringe. A constant voltage was applied to the sensor, and current was measured by a digital picoammeter. Air was allowed to pass into the glass dome after every ethanol gas exposure cycle.

## 3. Results and Discussion

When reactants were heated at 180 °C the reaction proceeded by the mechanism indicated in equation number 1 and 2 give the final product  $\text{LaFeO}_3$ .



The XRD measurement (Fig. 1) shows that the product is pure perovskite oxide  $\text{LaFeO}_3$  with an orthorhombic structure, the diffraction data are in good agreement with JCPDS card of  $\text{LaFeO}_3$  (JCPDS no: 15-0148). The average crystalline size of  $\text{LaFeO}_3$  perovskite powder is determine with the help of Scherrer's equation  $t = 0.9\lambda / \beta \cos\theta$ , where  $t$  is the average size of the particles,  $\lambda$  is wavelength of X-ray

radiation,  $\beta$  the full width at half maximum of the diffracted peak and  $\theta$  is the angle of diffraction [27-28]. The average crystalline size obtained for nanocrystalline  $\text{LaFeO}_3$  calcinated at  $1000^\circ\text{C}$  is found to be 28-63 nm.  $\text{LaFeO}_3$  nanocrystals are more attractive in the field of catalytic application. Glycine nitrate was used as organic fuel for combustion. Fig.1 shows desired and expected peaks of nanoporous  $\text{LaFeO}_3$ . No extra peaks are seen. Fuel plays an important role in the combustion of gel. At the combustion point organic fuel formed  $\text{NO}_x$ ,  $\text{CO}_2$ ,  $\text{N}_2$  gases and they tried to come out from the gel by breaking the gel into nanoporous and nanosized particles of  $\text{LaFeO}_3$ .

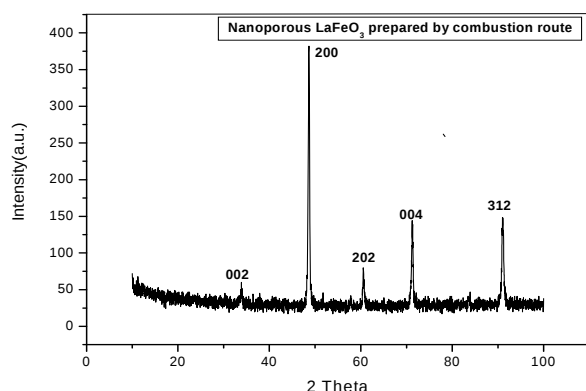


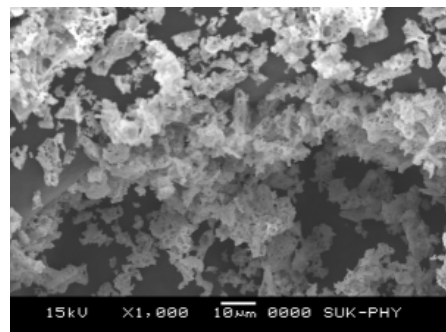
Fig. 1. XRD pattern of nanoporous  $\text{LaFeO}_3$ .

The SEM technique was employed for finding morphology of  $\text{LaFeO}_3$  powder heated at  $1000^\circ\text{C}$  Fig. 2 (a-d). One can notice the presence of macro-agglomerations of very fine particles having size less than  $1\ \mu\text{m}$ . The particle shapes are not well defined. Many large and small pores are present in the whole material. We assumed that the pores are mainly intergranular because intragranular pores are not seen on the SEM photograph.

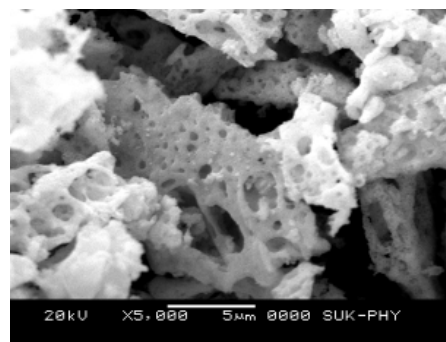
### 3.1. TEM Analysis

The TEM specimens were prepared by placing microdrops of colloid solutions on a carbon film supported by a copper grid. The TEM images of the nanocrystalline  $\text{LaFeO}_3$  calcinated at  $1000^\circ\text{C}$  in air for 5 h are shown in Fig. 3 (a) it indicates the presence of  $\text{LaFeO}_3$  nanoparticles with 28-63 nm size which form spherical type of oriental aggregation, agglomeration and polymeric linkage throughout the region. The HRTEM image Fig. 3 (b-c) shows the porous nature of the material. The pores are well developed spherical shape with diameter from 9-13 nm size. The results are correlated with the XRD. The selected area diffraction (SAED) pattern Fig. 3 (d) shows the spot type pattern which is indicative of single crystalline particles. No evidence

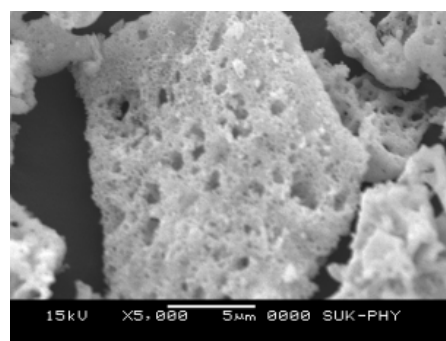
was found for more than one pattern, suggesting the single phase nature of the material. The BET surface area is measured on a Benchman coulter SA 3100 plus instrument using nitrogen adsorption at  $200^\circ\text{C}$ . The surface area of the synthesized powder is  $15.5\ \text{m}^2/\text{g}$ .



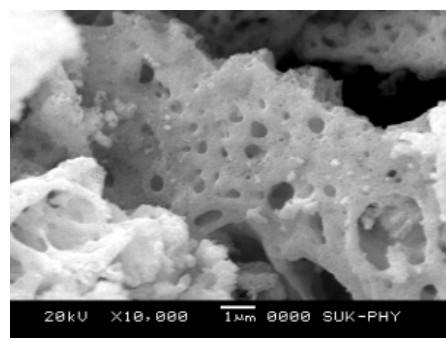
(a)



(b)

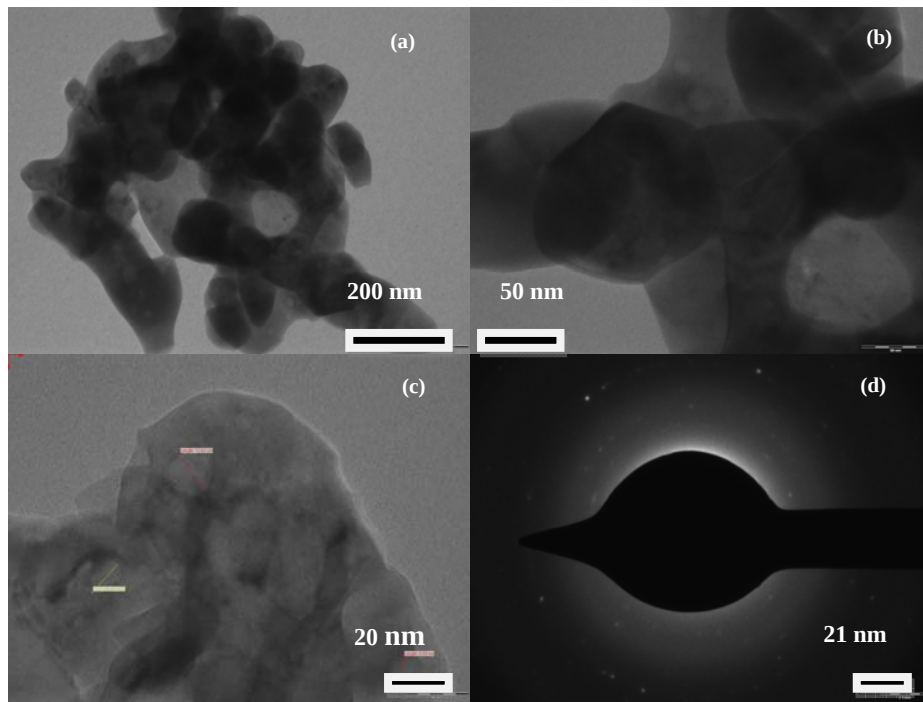


(c)



(d)

Fig. 2. SEM micrographs for  $\text{LaFeO}_3$  at  $1000^\circ\text{C}$ .



**Fig. 3.** TEM image of LaFeO<sub>3</sub> calcinated at 1000 °C (a), high resolution transmission electron microscopy image of nanocrystalline LaFeO<sub>3</sub> (b-c), selected area electron diffraction pattern (d).

### 3.2. Thickness Measurement

The thicknesses of the films were observed to be in the range from 30 to 40 μm. The reproducibility of the film thickness was achieved by maintaining the proper rheology and thixotropy of the paste.

### 3.3. Thermoelectric Power Measurements

The p-type semiconductivity of thick films of LaFeO<sub>3</sub> was confirmed by measuring thermoelectromotive force of the thick film samples. The LaFeO<sub>3</sub> was observed to be p-type material.

## 4. Electrical Properties

### 4.1. I-V characteristics

Fig. 4 depicts the I-V characteristics of the LaFeO<sub>3</sub> films at room temperature. It was clear from the I-V characteristics that the sliver contacts fabricated on the film were ohmic in nature. The voltage applied was in the range 1–30 V.

### 4.2 Electrical Conductivity

The semiconducting nature of LaFeO<sub>3</sub> is observed from the measurements of resistivity with temperature. The semiconductivity in LaFeO<sub>3</sub> must be due to large oxygen deficiency in it. The material

would then adsorb the oxygen species at higher temperatures ( $O_2^- \rightarrow 2O^- \rightarrow O^{2-}$ ). The increase in conductivity with increasing temperature could be attributed to negative temperature coefficient of resistance and semiconducting nature of the LaFeO<sub>3</sub>. It is observed from Fig. 5 that the electrical conductivity of the LaFeO<sub>3</sub> in air is nearly linear in the temperature range from 300–575 °K in air ambient.

## 5. Sensing Performance

### 5.1. Sensing Characteristics

The relative response to a target gas is defined as the ratio of the change in conductance of a sample upon exposure of the gas to the original conductance in air. The gas response can be written as:

$$\text{Gas Response} = \frac{G_g - G_a}{G_a} = \frac{G}{G_a},$$

where  $G_a$  is the conductance in air and  $G_g$  is the conductance in a sample gas. Specificity or selectivity can be defined as the ability of a sensor to respond to a certain gas in the presence of different gases. Response time (RST) was defined as the time required for a sensor to attain 90 % of the maximum increase in conductance after the exposure of test gas on the sensor surface, while recovery time (RCT) as the time taken to get back 90 % of the maximum conductance in air.

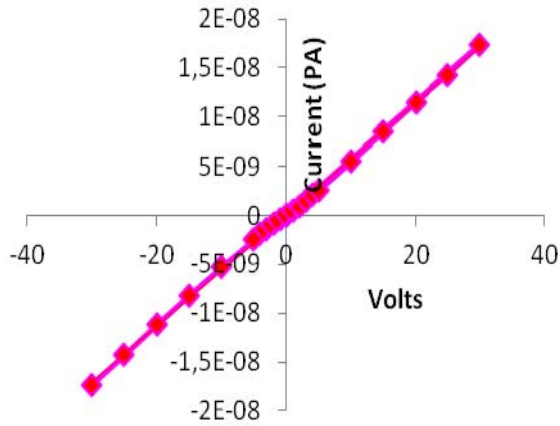


Fig. 4. I-V characteristics OF  $\text{LaFeO}_3$ .

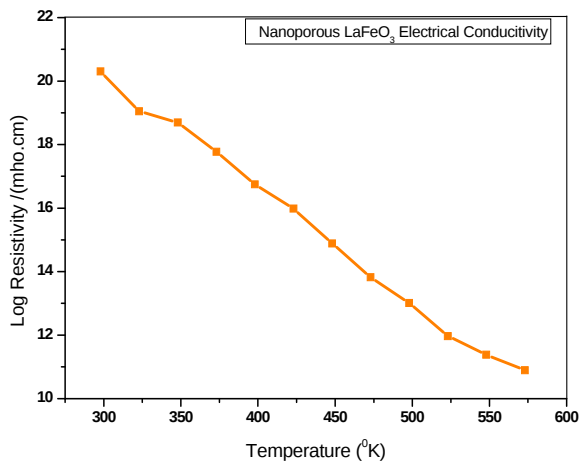


Fig. 5. Electrical conductivity of  $\text{LaFeO}_3$  sample in air.

### 5.3. Gas Response of $\text{LaFeO}_3$ at Various Temperatures

#### 5.3.1. Gas Response and Ethanol Gas Concentration

The variation of response of  $\text{LaFeO}_3$  sample with ethanol gas concentration is represented in Fig. 6. It is clear from the figure that the gas response goes on increasing with gas concentration up to 100 ppm at 300 °C. The rate of increase in response was relatively larger up to 100 ppm and smaller beyond 100 ppm. Thus, the active region of the sensor would be up to 100 ppm.

#### 5.3.2. Gas Response and Operating Temperature

Fig. 7 represents the variation of ethanol gas response with the operating temperature of the  $\text{LaFeO}_3$  film. It is clear from the figure that the gas response increase with operating temperature reaches to the maximum (100 ppm) at 300 °C, and falls with further increasing

the in operating temperature. The ethanol may burn before reaching the surface of the film at higher temperatures (>300 °C). Hence, the gas response would have been decreased above 300 °C. A larger amount of oxygen-adsorption would have occurred on the surface of the film at 300 °C and have facilitated the sensor to oxidize the ethanol gas immediately, giving faster and larger gas response.

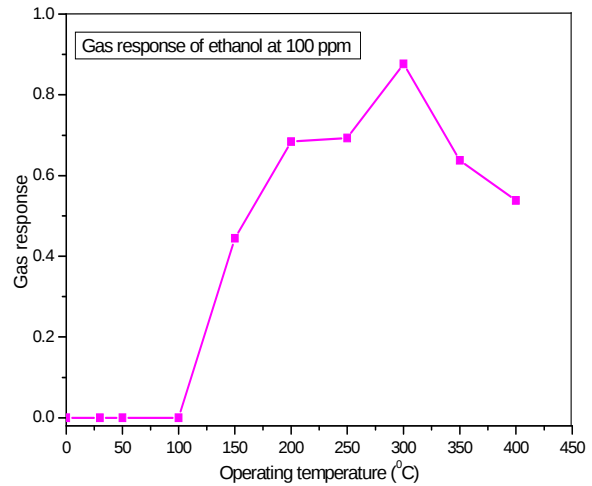


Fig. 6. Variation of response with operating temperature.

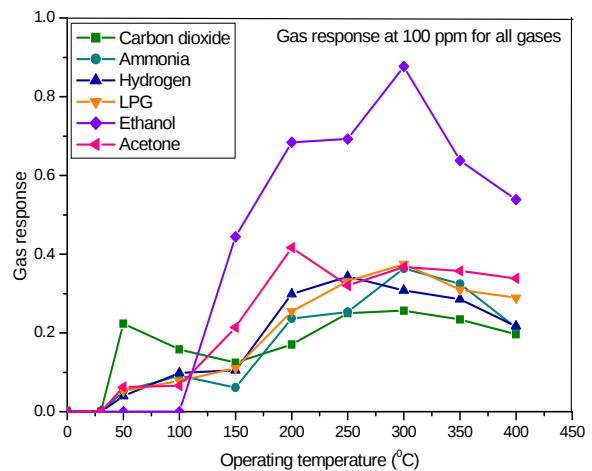


Fig. 7. Variation of gas response with operating temperature.

#### 5.3.3. Ethanol Gas Response and Gas Concentration

Fig. 8 represents the variation of ethanol gas response with the gas concentration of  $\text{LaFeO}_3$  film. For  $\text{LaFeO}_3$  film, the response values were observed to increase continuously with increasing the gas concentration up to 100 ppm at 300 °C. The rate of increase in response was relatively larger up to 100 ppm, and then saturated after 100 ppm. Thus, the active region of the sensor would be between 50 and 100 ppm.

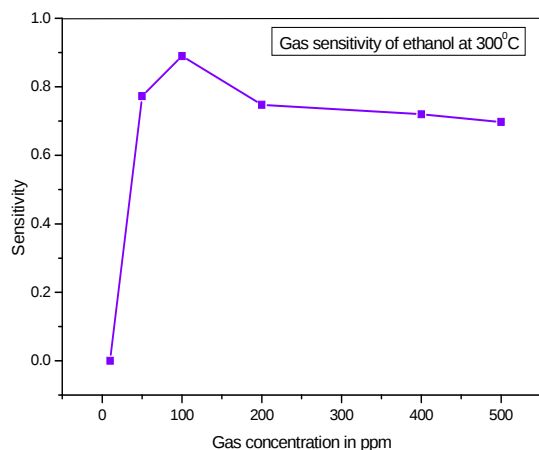


Fig. 8. Variation of gas response with gas concentration.

### 5.3.5. Selectivity for Ethanol Against Various Gases

Fig. 9 depicts the selectivity of  $\text{LaFeO}_3$  to 100 ppm of ethanol gas against various gases at 300 °C. It is clear from Fig. 9 that, in contrast to pure  $\text{LaFeO}_3$ ; the sample shows not only enhanced response towards ethanol but also very high selectivity.

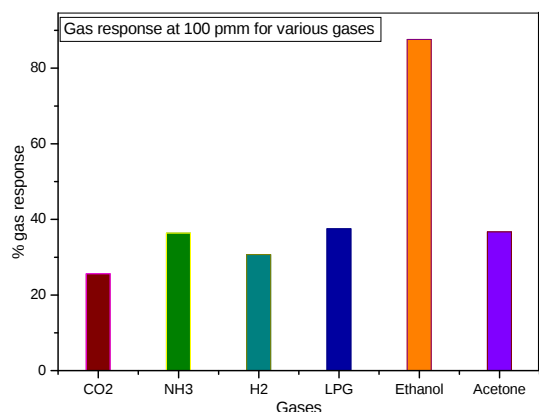


Fig. 9. Gas responses among various gases.

### 5.3.6. Response and Recovery of the Sensor

The response and recovery of the  $\text{LaFeO}_3$  sensor are represented in Fig. 10. The response was quick (~5 s) to 100 ppm of ethanol. Recovery was also very fast (~35 s). The fast response may be due to the fast oxidation of ethanol into  $\text{H}_2\text{O}$  (gas). After sevaporing water molecules, the film recovery was obtained.

## 7. Summary

The following statements can be made for the sensing performance of  $\text{LaFeO}_3$  sensors:

- (1) Pure  $\text{LaFeO}_3$  thick film is the most sensitive element to ethanol gas;
- (2) The optimum operating temperature for ethanol gas sensing is 300 °C;
- (3)  $\text{LaFeO}_3$  has the potential of fabricating ethanol sensor;
- (4) Surface properties of the films could be conveniently customized (without affecting the bulk properties) by the surface modification using dipping technique;
- (5) The  $\text{LaFeO}_3$  thick film sensor showed very rapid response and recovery to ethanol gas;
- (6) The sensor showed good selectivity to ethanol gas against LPG,  $\text{NH}_3$ ,  $\text{CO}_2$  and  $\text{H}_2$  gases.

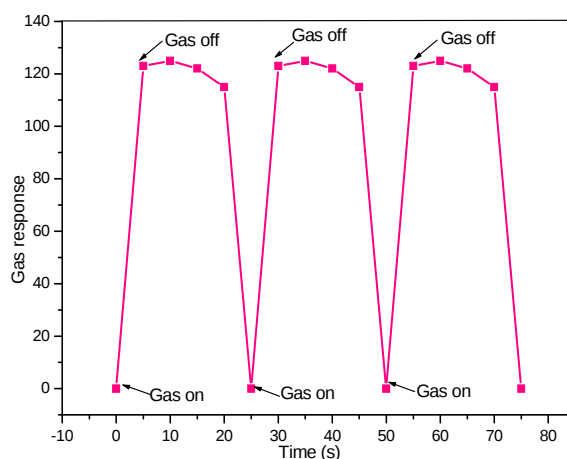


Fig.10. The response and recovery of the  $\text{LaFeO}_3$  sensor.

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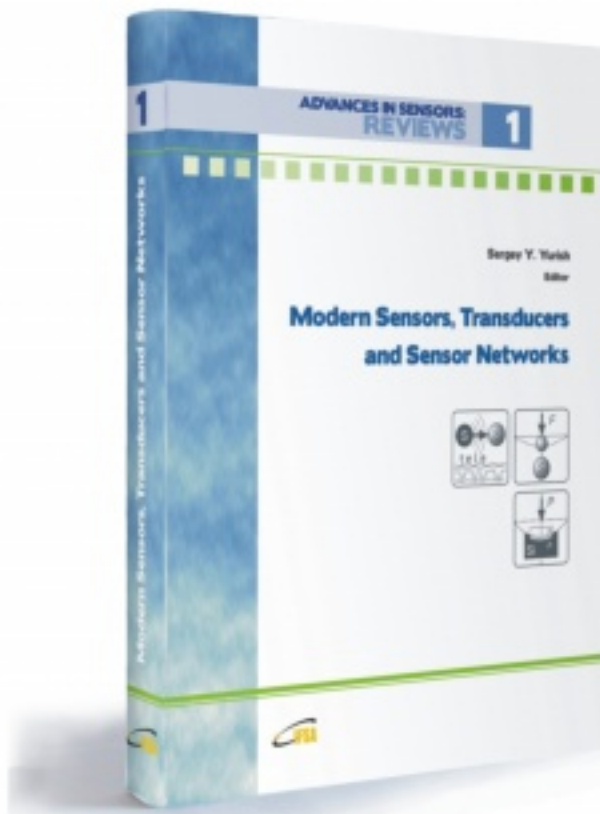
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