

The Effect of Couple Doping Gd and Co on The Physical Characteristics of LaFeO₃ Thick

Film for Acetone Gas Sensor Application

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Abstract

The acetone gas sensor is one type of sensor being researched for its application because it detects the presence of diabetes in sufferers. Gas sensors with high sensitivity and low operating temperature have been extensively investigated for this purpose, and this research is focused on the same purpose. Synthetization and characterization of LaFeO₃ with co-doping Gd₂O₃ and CoO thick film ceramics for acetone gas sensor was conducted. LaFeO₃ was made using the co-precipitation method with 2.5% CoO for each and 0%, 2.5%, and 5% Gd₂O₃ variation to the LaFeO₃. The LaFeO₃ thick film was prepared using the screen-printing technique and calcined at 800°C for two hours. The analysis of crystal structure characterization using X-Ray Diffraction (XRD) resulted in LaFeO₃ with co-doping Gd₂O₃ and CoO thick film ceramics having the same cubic crystal phase with smaller lattice parameters and crystallite sizes after doping were added. The results of morphology structure characterization using Scanning Electron Microscopy (SEM) showed the grain size of the LaFeO₃ with co-doping 2.5% CoO and 0%, 2.5%, and 5% Gd₂O₃ samples to support the analysis of electric property characterization later on. The electric property characterization showed that LaFeO₃ with various Gd₂O₃ concentrations, as part of co-doping with 2.5% CoO, resulted in higher sensitivity compared to the lacking of Gd₂O₃ one. In order, the maximum sensitivity values of each Gd₂O₃ concentration are 2.74, 3.06, and 8.76 when exposed to 270 ppm acetone gas at 310°C. Gd₂O₃, as part of co-doping in LaFeO₃ with CoO 2.5%, has successfully increased the sensitivity to the gas sensor yet still can not meet the expectation towards the operating temperature, which is still high compared to other references.

Keywords: LaFeO₃; Gd₂O₃; CoO; Thick Film; Acetone Gas Sensor

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INTRODUCTION

The gas sensor is a sensor that is used to detect and identify the presence of gas in an area [1]. The acetone gas sensor is one type of sensor being researched for its application to detect the presence of diabetes in sufferers [2]. In general, the materials used for manufacturing gas sensors are oxide semiconductors and metal oxides [3]. Some examples of materials used include SnO₂ [4], TiO₂ [5], Fe₂O₃ [6], LaFeO₃ [7], and so forth [8].

LaFeO₃ is one of the constituent materials of gas sensors, which is sensitive to certain gases [9-11]. However, to be able to detect the presence of gas in a large range, it is necessary to engineer the material by doping [12]. The doping carried out by Chen et al. on La_{1-x}Pb_xFeO₃ (x = 0.0-0.5) has a sensitivity of 7 for 50 ppm acetone gas [13]. Pb doping causes the average grain size to be smaller, and the effect on the surface to volume ratio becomes larger so that the gas sensor performance increases [13,14]. Hao et al. also doped Ni on LaFeO₃, so a sensitivity of 82.75 was obtained for 100 ppm acetone gas [15]. Smaller grain size after doping increases the sensitivity of the gas sensor [12,16]. However, the operating temperature of the gas sensor is still relatively high for the two previous studies, which are 240°C [13] and 220°C [15], respectively.

Co-doping is the distribution of two dopants, each of which is a different element, to improve the performance of its electronic properties [17]. Gd was chosen as one of the dopants because several studies showed that Gd as a dopant increased the sensitivity in terms of the structure after being doped [18] with low concentrations for gas sensor applications [19,20]. In addition, Gd also provides a lower operating temperature than the two previous studies, namely 200°C for acetone gas [18]. The element Co was chosen as the Gd dopant partner in the co-doping of this study because a study using CoO has shown good performance in detecting the presence of acetone gas with high sensitivity [21]. Couple doping has shown promising results in terms of enhancing material properties, especially in lowering operating temperature and gas detection sensitivity, which is proven by Zeany et al. focusing on LaFeO₃ couple doped with ZnO and CaO [22].

With that knowledge to enhance sensitivity and lower operating temperature, this study's objective would focus on manufacturing acetone LaFeO₃ gas sensors with Gd and Co dopant in couple doping (co-doping), hoping to achieve both aspects.

METHOD

Preparation of LaFeO₃ co-doping Gd and Co Powders

In this study, three samples of LaFeO₃ were being couple-doped with Gd₂O₃ as much as 0%, 2.5%, and 5% mole percent, along with CoO for each sample of 2.5% mole percent on La₂O₃, Fe₂O₃, Gd₂O₃, and CoO powder were being made using co-precipitation technique. Each powder was dissolved in 12 M HCl and mixed to form a mixture of LaFeO₃ co-doping Gd and Co. The mixture was precipitated using ammonia hydroxide and then filtered and dried at 100°C for 6 hours and calcined in a furnace for 3 hours at 800°C.

Thick Film Fabrication

The calcined material, which is already in powder form, is made in the form of a thick film using the screen-printing technique. Each powder is mixed with an organic vehicle (OV) with a composition ratio of 70% OV and 30% powder to form a paste. The paste was coated on an alumina substrate that had been coated with silver and burned at 800°C for two hours.

Thick Film Characterization

Structural characterization was done using X-Ray Diffraction (XRD) Rigaku Smart Lab for crystallography in Institut Teknologi Bandung's Hydrogeology and Hydrogeochemistry Laboratory with 2theta (2θ) ranging from 20° to 80° and Scanning Electron Microscopy (SEM) JSM-6510A type with the sample needs to be dry with 1 cm diameter and 5mm thick in maximum. XRD was carried out to see the crystal structure of thick film ceramics, and the results were analyzed using *Match!3*, so the crystal phase, crystal plane orientation (miller index, hkl), lattice parameters (a , b , and c) and crystallite size (D) were obtained. The crystallite size was obtained from the Debye-Scherrer Equation [23], which is shown in Equation (1), namely

$$D = \frac{0.9\lambda}{B \cos\theta} \quad (1)$$

SEM was carried out to see the morphological structure of thick film ceramics. The results were analyzed by comparing the resulting image scale with the standard magnification scale to obtain the grain size.

Characterization of the electrical properties was conducted to see changes in the gas sensor when exposed to acetone gas, which was indicated by changes in resistance and sensitivity. This characterization was carried out using a gas chamber tool set. The electrical characterization test was carried out by measuring the resistance of the sample to temperatures up to 315°C by recording data for every 5°C change, both in ambient conditions and when acetone gas was given with varying concentrations, starting from 90 ppm, 180 ppm, and 270 ppm. The data from the resistance test results are in the form of the resistance value and the temperature when the resistance is read. The data is then plotted against temperature to see changes in resistance and then see how sensitive it is. The sensitivity value is obtained from Equation (2) [24].

$$S = \frac{(R_g - R_a)}{R_a} \quad (2)$$

RESULTS AND DISCUSSION

Crystal Structure Characteristic

The results of the crystal structure characterization in the form of XRD test are presented in the form of a graph of the diffraction pattern shown in Figure 1. The LaFeO_3 diffraction pattern appears to have formed along with Fe_2O_3 and alumina substrate, based on the comparison with the reference database from the Crystallography Open Database no. 96-154-2033 [25]. The crystal phase formed for the three samples has the same phase, namely a $P\bar{m}3m$ cubic crystal phase with miller index in sequential order (100), (110), (111), (200), (210), (211), (220), and (310). The lattice parameters were obtained using the relation between Bragg's law and $h^2 + k^2 + l^2$'s cubic structure. LaFeO_3 with co-doping Co 2.5% with Gd 0% has the value of $a = b = c = 3.9301 \text{ \AA}$, for Gd 2.5% it is $a = b = c = 3.9284 \text{ \AA}$, and for Gd 5%, it is $a = b = c = 3.9274 \text{ \AA}$.

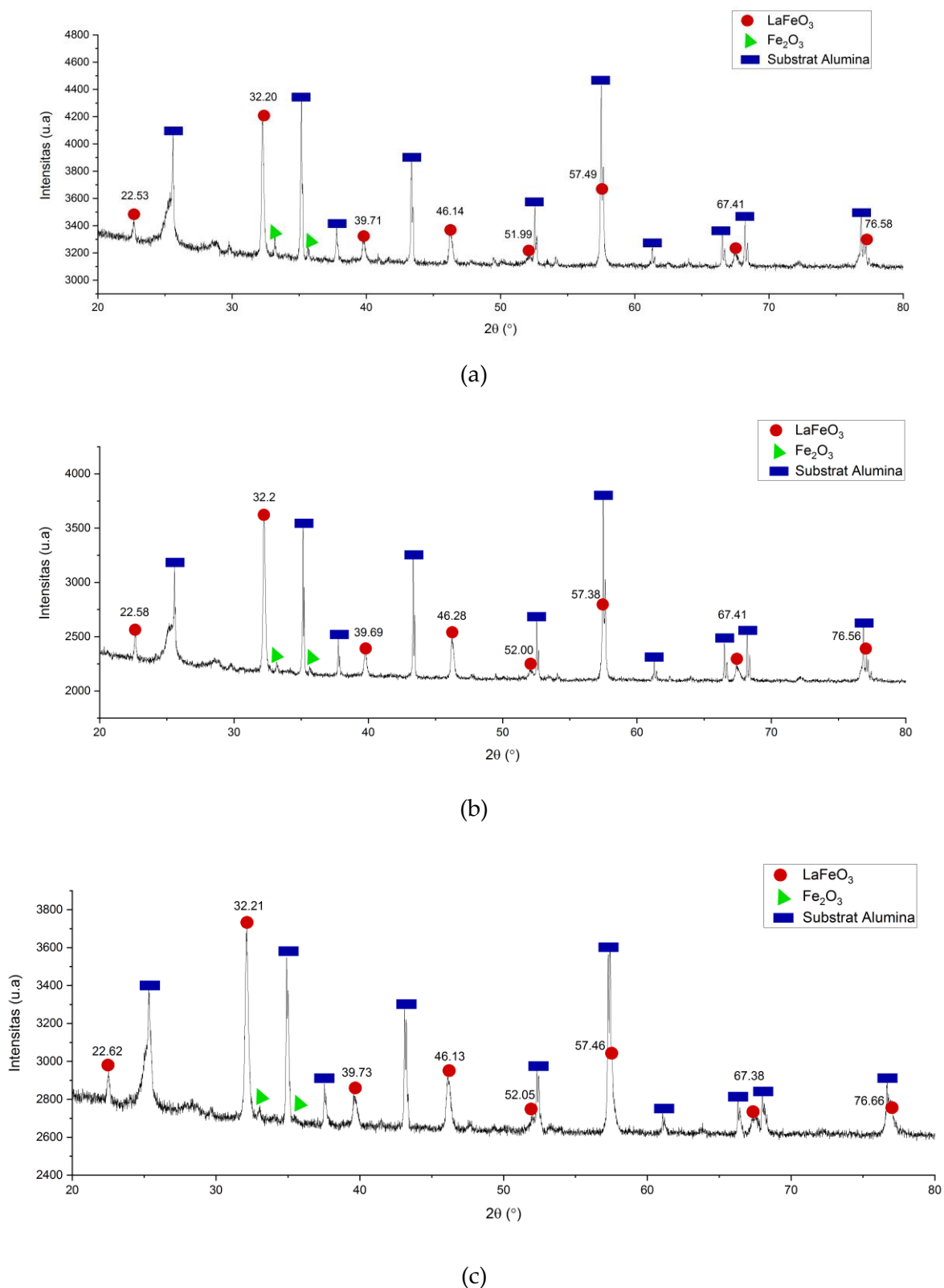


Figure 1. The Result of XRD Characterization of LaFeO₃ co-doping Gd and Co 2.5% with Gd (a) 0%, (b) 2.5%, and (c) 5%.

The reference from the Crystallography Open Database (COD) database no. 96-154-2033, shows that the LaFeO₃ lattice parameter has the value of $a = b = c = 3.9260 \text{ \AA}$. In comparison, they are not very different from the reference. This fact shows that the Gd³⁺ ion from Gd₂O₃ and Co²⁺

ions from CoO have succeeded in substituting each La^{3+} ion from La_2O_3 and Fe^{3+} from Fe_2O_3 . Gd^{3+} has an ionic radius of 1.07 Å, while La^{3+} has an ionic radius of 1.17 Å. Besides, Co^{2+} has an ionic radius of 0.79 Å, while Fe^{3+} has an ionic radius of 0.69 Å. Co^{2+} ion substitution on Fe^{3+} on each sample gives a LaFeO_3 lattice parameter value greater than the reference. When Gd started to get in Gd^{3+} , which has an ionic radius smaller than La^{3+} , substituting La^{3+} ions, thus causing the lattice parameters to decrease as the Gd doping concentration is added [25].

The crystallite size decreases as the Gd doping concentration increases but expands at 2.5% Gd, which starts from 64,335 nm, 68,839 nm, to 65,752 nm. The crystallite size was calculated with the Debye-Scherrer Equation shown in Equation (1) [26].

Morphology Structure Characteristic

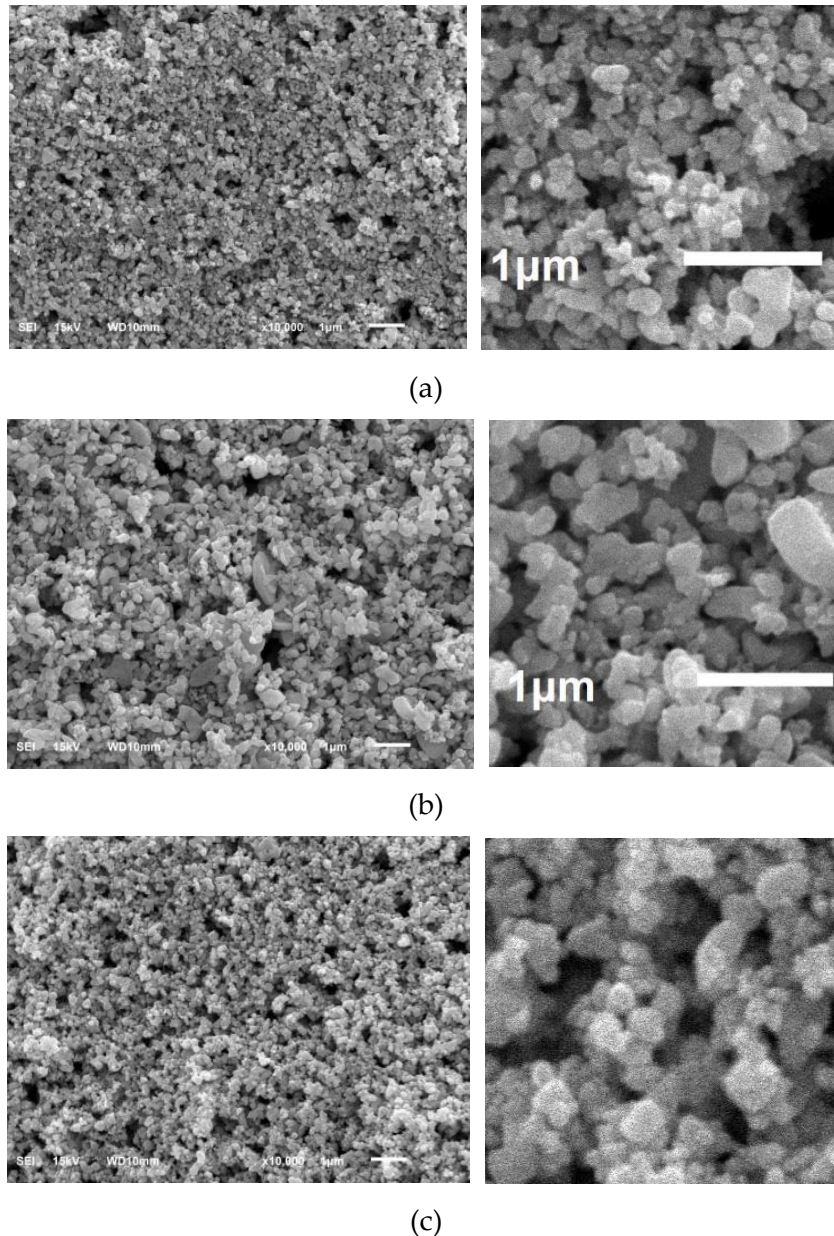


Figure 2. The Result of SEM Characterization of LaFeO_3 co-doping Gd and Co 2.5% with Gd (a) 0%, (b) 2.5%, and (c) 5%.

The results of the morphological structure characterization in the form of sample grain size

from the Scanning Electron Microscopy (SEM) test results are shown in Figure 2. The SEM test results on the thick film LaFeO₃ co-doping Co 2.5% with Gd 0%, 2.5%, and 5% indicate the average grain size in sequential order are 0.15 μm, 0.26 μm, and 0.16 μm which calculated using linear intercept method. Adding Gd 2.5% to the sample causes the average grain size to be larger than that of Gd 0%. The thick film LaFeO₃ co-doping Co 2.5% with Gd 5% has an average grain size smaller than Gd 2.5% but larger than Gd 0%. This phenomenon is in line with the crystallite size from the XRD test results, which shows that the crystallite size increases when 2.5% Gd is added. In addition, the grain sizes in this sample have a more uniform size with each other, compared to the other two samples.

Seeing the size of crystallite size in XRD results, with the same trend, the grain will increase in size after heat treatment and decrease after the next doping [5]. This fact might be because the doping didn't form very well with LaFeO₃, thus increasing the grain boundaries, so it didn't grow as much as the previous doping. This behavior happened in Neng Astri et al.'s research [24].

Electrical Properties Characteristic

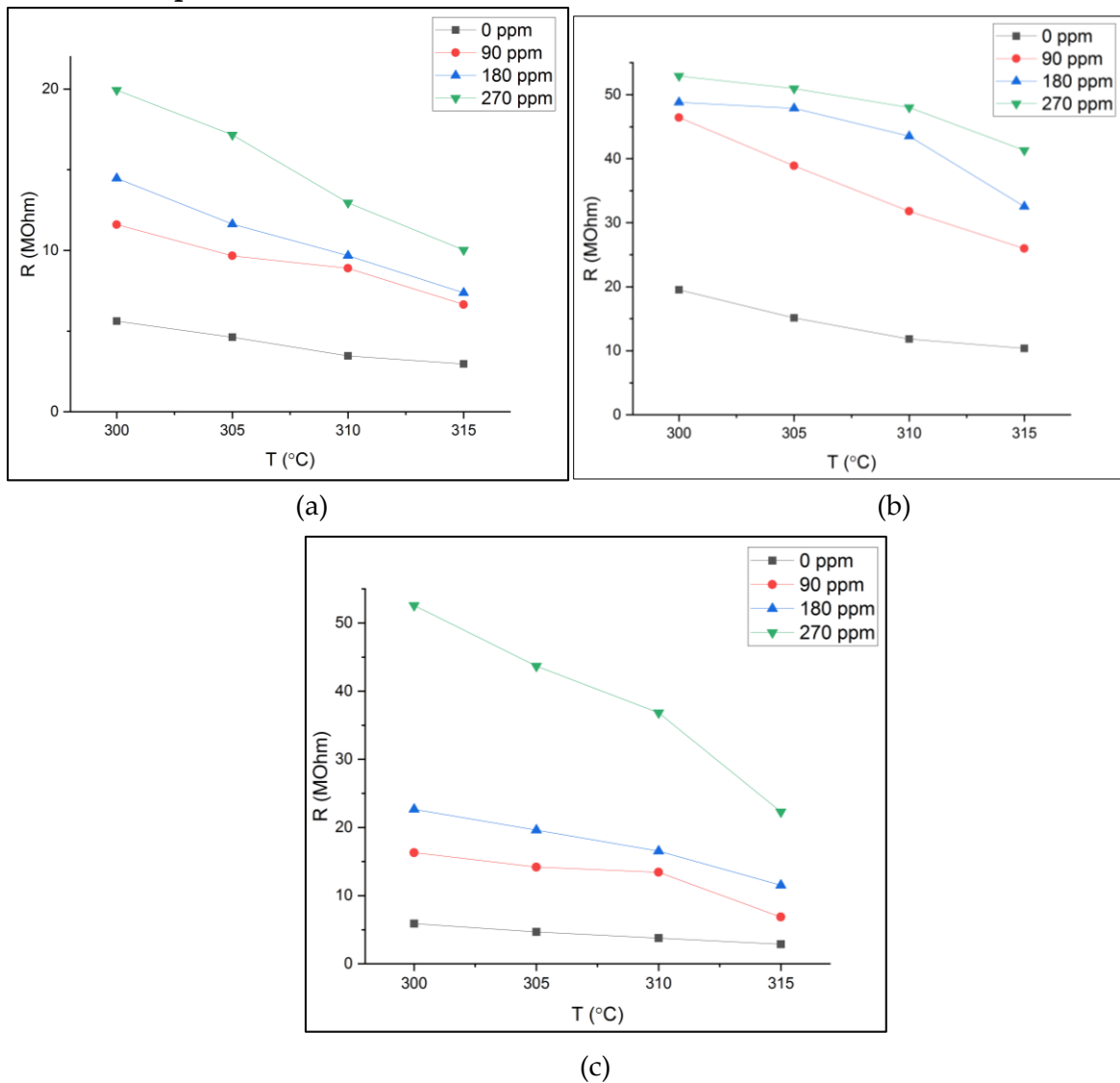


Figure 3. The effect of temperature on LaFeO₃ co-doping Gd and Co 2.5% thick film ceramic resistance with Gd (a) 0%, (b) 2.5%, and (c) 5%.

The results of the sample resistance measurement to temperature for each sample show that the resistance decreases as the temperature increases, as shown in Figure 3. The decrease in resistance value with increasing temperature is caused by ionized electrons from the valence band. The ionized electrons will leave holes in the valence band [27]. The higher the temperature, the more electrons are ionized, and the more holes are produced. The presence of this hole gives a decrease in the resistance value [28].

The results of the sample resistance measurement to temperature can also be used to find the sensitivity value for each sample [29] and determine the operating temperature of the gas sensor. This procedure is done using Equation (2), and the trend of temperature changes with sensitivity is shown in Figure 4.

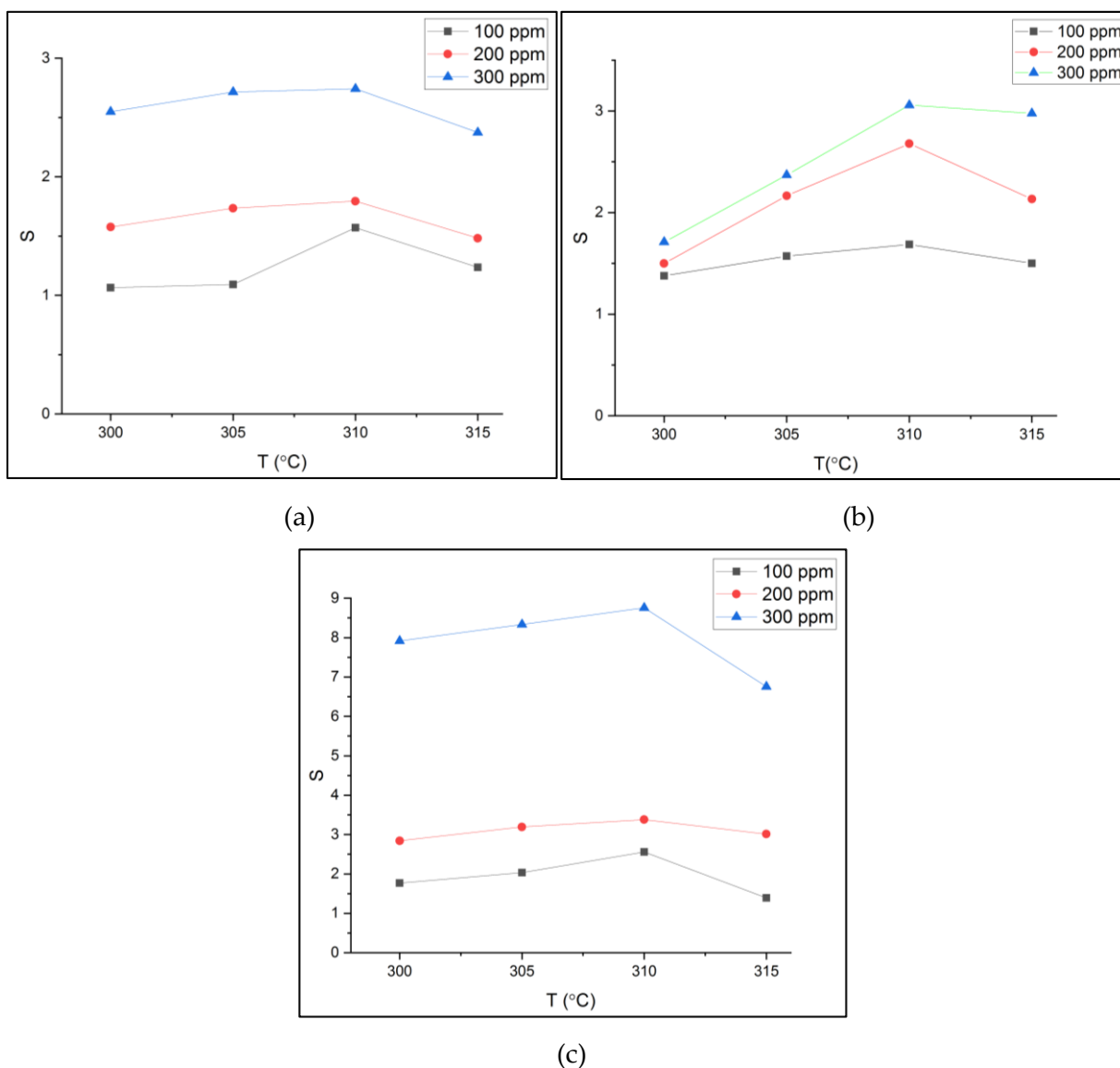


Figure 4. The effect of temperature on LaFeO₃ thick film ceramic sensitivity with (a) 0% Gd, (b) 2.5% Gd, and (c) 5% Gd.

Adding Gd doping to LaFeO₃ Co 2.5% affects the sensitivity value obtained. In addition, the concentration of gas that is used also has an influence on the sensitivity value obtained. The sensitivity increases with increasing Gd doping and the concentration of acetone gas being used

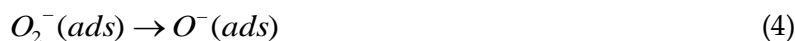
[19,30,31]. More detailed data is depicted in Table 1.

Table 1 The sensitivity and operating temperature of LaFeO₃ co-doping Co 2.5% with Gd 0%, Gd 2.5%, dan Gd 5% thick film ceramic.

Compounds	Operating Temperature (°C)	S		
		90 ppm	180 ppm	270 ppm
LaFeO ₃ Co 2.5% Gd 0%	310°C	1.570199	1.794661	2.741911
LaFeO ₃ Co 2.5% Gd 2.5%		1.686386	2.677766	3.057973
LaFeO ₃ Co 2.5% Gd 5%		2.557392	3.379999	8.758197

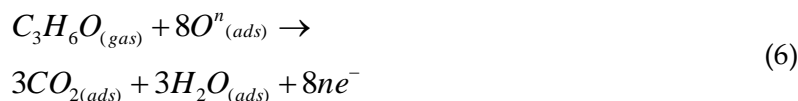
The sensitivity value is obtained from the highest peak value on the sensitivity graph to the operating temperature, while the operating temperature is the temperature that provides the highest sensitivity [32]. Based on the data in Table 1, the operating temperature for the LaFeO₃ Co 2.5% thick film for each concentration of Gd has the same value, which is 310°C.

When the sensor surface is exposed to free air, the surface of the LaFeO₃ material adsorbs oxygen. Oxygen molecules attract electrons from the surface of LaFeO₃ to form adsorbed oxygen. When the temperature is increased, the adsorbed oxygen undergoes the following reaction [27,28]:



In this state, the electrons in the valence band gain thermal energy for ionization to the conduction band until they are captured by oxygen, leaving holes in the valence band. These holes' presence lowers the material's resistance [13]. The more oxygen is adsorbed, the more electrons are captured [33], and the more holes are formed.

When exposed to acetone gas, acetone gas will attract oxygen with the consequence that electrons in oxygen will be released on the material surface [34]. The possible reaction on the LaFeO₃ sensor can be explained by the equation (6):



The electrons that come will lose energy, so they will fall into the valence band where there is a hole [32]. Electrons will meet holes, and recombination occurs.



During recombination, electron-hole pairs are lost as charge carriers. The more electrons, the more recombination, and loss of holes as charge carriers. These phenomena cause the resistance value to be high [13,14]. This fact corresponds to the resistance value when the sample is exposed to gas [35]. Thus, from the measurement results of electrical properties, it is shown that LaFeO₃ co-doping Co 2.5% with Gd 0%, 2.5%, and 5% is a p-type semiconductor which is characterized by a smaller resistance value when without gas compared to when being exposed

to the gases [36].

As long as the temperature is continuously increased, the reaction of the adsorbed oxygen, both with the material's surface and with acetone gas, will continue to 310°C. When passing through it, the hole production will decrease drastically, which is indicated by reduced sensitivity. This result is because there are myriads of electron holes that recombine. [37].

From the crystal structure perspective, XRD results show that LaFeO₃ co-doping with CoO and Gd₂O₃ has been successfully achieved. In addition, from the sensitivity value, it is found that given doping concentration increases the sensitivity. This fact is supported by the SEM results showing grain sizes that are uniformly increasing to each other after doping was added. The grain sizes are distributed and more uniform, increasing the sensor's surface ratio, thus enabling the sensor to have high sensitivity [33].

However, compared with the previous references like by Hao et al. [15] and Zhang et al. [13], operating temperatures show a higher value. This fact is suspected from the synthesis process results until it becomes a paste that is not maximized. The addition of doping which is expected to lower the operating temperature has not been achieved. This result is supported by the results of the SEM test, which showed the morphological structure of sensors that have been made. However, for sensitivity, it has a more significant value compared to the study conducted by Zhang et al. [13]

This research could've done better with standardized experiment apparatus because this is actually the only limitation that this research faces. Besides, further studies can focus on the time response and higher range of doping that can be added to the sample, thus can make this research more appropriate scientifically to consider.

CONCLUSION

The synthesis and characterization by co-precipitation method on thick film ceramics LaFeO₃ co-doping Gd₂O₃ and CoO for acetone gas sensor applications have been carried out. The crystal structure of the LaFeO₃ thick film ceramics co-doping Co 2.5% with Gd 0%, Gd 2.5%, and Gd 5%, each of which has a cubic phase with the crystallite size decreasing as Gd doping concentration increases. The grain size increases when the sample is given Gd 2.5% and again decreases when given Gd 5%. The gas sensor with the addition of Gd 5% concentration has a higher sensitivity than the gas sensor with Gd 0% and 2.5% Gd. In addition, for each sample with the concentration of acetone gas tested, the operating temperature is shown to be 310°C. The high sensitivity after a couple of doping is achieved compared to the references. However, for the operating temperature, it shows a higher value when compared with the previous research reference. The crystallite size from XRD results and SEM results support it.

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AUTHOR CONTRIBUTIONS

Hendi Haryadi: Methodology, Formal Analysis, Investigation, and Writing - Review & Editing; Dani Gustaman Syarif: Conceptualization, Validation, Formal Analysis, and Supervision; and Endi Suhendi: Conceptualization, Formal Analysis, Writing - Original Draft

and Review & Editing.

DECLARATION OF COMPETING INTEREST

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.

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