EFFECT OF FILM THICKNESS ON ELECTRICAL CHARACTERISTIC OF Fe₂O₃ THICK FILM CERAMICS MADE FROM LOCAL MINERAL IN AIR AND ETHANOL ATMOSPHERE

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Abstract. In order to find possibility to utilize local mineral for high value products, fabrication of Fe₂O₃ thick film ceramics with thickness variation from local mineral of yarosite has been carried out and characteristics especially electrical characteristics of the thick film ceramics have been studied. Powder Fe₂O₃ was derived from yarosite using precipitation method. The nano powder of Fe₂O₃ was mixed with organic vehicle (OV) consists of alpha terpineol and ethyl cellulose to form a paste. The paste was screen printed on alumina substrates using screen sizes of 183 mesh, 225 mesh and 375 mesh. The green thick films were fired at temperature of 950°C for 90 minutes. The fired film was analyzed using an x-ray diffractometer (XRD) to know its crystal structure and a Scanning Electron Microscope (SEM) to know its morphology. Electrical resistance of the thick films was measured at various temperatures in air and air containing ethanol gas. Visual appearance of fired thick film showed that the films have been well produced. The XRD data showed that the thick films had phase of hematite. The SEM data showed that the thick films were porous with relatively small grain size. The electrical resistance of the thick films increased as the increase of film thickness. The electrical resistance in air was higher than that of the thick films in air containing ethanol gas indicating the thick films had potentiality for ethanol gas sensor application.

Keywords: Thick film, Fe₂O₃, ethanol, gas sensor

1 Introduction

In order to get high economical value of mineral that is abundant in Indonesia, it is required to convert it into high value product such as gas sensor. Gas sensor can be made from many semiconductive ceramics such as $MgFe_2O_4[1]$, ZnO [2] and $Fe_2O_3[3]$ which can be derived from mineral. In order to find possibility to utilize local mineral for gas sensor, here, fabrication of Fe_2O_3 thick film ceramics from local mineral of yarosite was done.

Thick film ceramics were fabricated using screen printing technique. Characteristics especially electrical characteristic of the thick films depend on some parameters and one of them is screen size. The different size of screen will produce different thickness of the thick film. The different thickness may produce different characteristics of the films. Here, the effect of the different size of screen Effect of Film Thickness on Electrical Characteristic of ${\sf Fe_2O_3}$ Thick Film Ceramics Made from Local Mineral in Air and Ethanol Atmosphere

on the electrical characteristics of the thick film in air and air containing ethanol gas was studied and discused.

2 Methodology

Powder of Fe_2O_2 was prepared from yarosite mineral. The powders of yarosite was dissolved in HCl. A certain amount of NH4OH was added into the solution. A precipitate was formed. The precipitate was then dried and calcined at 700°C for 2 hours to get Fe_2O_3 powder. The powder was analyzed using x-ray diffraction (XRD). The powder of Fe_2O_3 was mixed with organic vehicle (OV) consisting 10 % ethyl clulose and 90 % terpineol to form a paste. The paste was screen printed on alumina substrates with different sizes of screen of 183, 225 and 375 mesh. Green thick films were fired at 950°C for 90 minutes. Crystal structure of the thick films was analyzed using XRD and morphology of them was analyzed using a scanning electron micoscope (SEM). Silver paste was screen printed on the surface of the thick films to form electrodes. Electrical resistance of the films was measured at different temperatures from about 50°C to 400 °C in air and ethanol gas atmospheres.

3 Results and Discussion

Synthesis of Fe₂O₃ powder

Figure 1 is visual appearance of Fe_2O_3 powder prepared by using precipitation method. Chemical composition of the powder is shown in Table 1. It can be seen that SiO_2 and TiO_2 are impurities with significant concentration compared to others. XRD profile of the Fe_2O_3 powder is shown in Figure 2. It is clearly seen that powder has crystal structure of hematite (JCPDS No. 33-0664). Particle size of the powder is 30 nm as calculated using Debye Scherrer method [4,5].

Tabel 1. Chemical composition of Fe₂O₃ powder prepared from mineral Yarosit.

No.	Substance	Concentration (%)
1.	Fe_2O_3	93,80
2.	SiO_2	1,02
3.	MgO	0,09
4.	CaO	0,19
5.	TiO_2	1,15
6.	MnO	0,12
7.	Na_2O	0,59
8.	K_2O	0,50



Figure 1. Visual appearance of $\overline{Fe_2O_3}$ powder prepared using precipitation method.



Figure 2. XRD profile of Fe₂O₃ powder prepared using precipitation method.

Fabrication and Characterization of Fe2O3 Thick Films

Visual appearance of the fired Fe₂O₃ thick film is shown in Figure 3, as a representative. The thick film is good without crack. Thickness of the thick films is 20 μ m (screen size of 375 mesh), 30 μ m (screen size of 225 mesh) and 60 μ m (screen size of 183 mesh), respectively.

The XRD profiles of the films are shown in Figure 4-6. As can be seen from the Figure 4-6, the crystal structure of all the films is the same, namely hematite (JCPDS No. 33-0664). Some peaks of alumina substrate (indicated with A) was found in the XRD profile of the film prepared with screen of 375 mesh. It is because the film is thinner than the other ones. This fact shows that the thickness of the film does not affect the crystal structure formation.



Figure 3. Visual appearance of the fired Fe_2O_3 thick film.

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Figure 4. XRD profile of Fe₂O₃ thick film fired at 950°C for 90 minutes with screen of 183 mesh.



Figure 5. XRD profile of Fe₂O₃ thick film fired at 950°C for 90 minutes with screen of 225 mesh.



Figure 6. XRD profile of Fe₂O₃ thick film fired at 950°C for 90 minutes with screen of 375 mesh.

SEM images of Figure 7 to 9 show that all of the films are porous with small grains (sub micron). The porosity increases as the screen size increases. The grain size is little bit larger as the increase of screen size although the increase is not significant.



Figure 7. SEM image of morphology of Fe₂O₃ thick film fired at 950oC for 90 minutes with screen of 183 mesh.



Figure 8. SEM image of morphology of Fe₂O₃ thick film fired at 950oC for 90 minutes with screen of 225 mesh.



Figure 9. SEM image of morphology of Fe₂O₃ thick film fired at 950oC for 90 minutes with screen of 375 mesh.

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Electrical Characteristic

Samples were designed according to Figure 10. Electrical characteristic of the Fe_2O_3 thick films in air and air containing ethanol gas is shown in Figure 11. One can see from Figure 11 that the electrical resistance of the film made using screen with size of 183 mesh is smaller than that of the film made using screen with size of 225 mesh and 375 mesh. The larger the screen size, the larger the electrical resistance of the film. It means that the thinner the film, the larger the electrical resistance. This tendency follows the relation between electrical resistance and bulk volume of the keramik for the same electrical resistivity. However, the electrical resistance change is influenced also by the porosity of the film ceramic.

Electrical resistance of the films in ethanol gas atmosphere is lower than that in air. This is caused by the interaction of the etahnol gas with the film ceramic. The ethanol gas transfers electron to the film ceramic, increasing electron in conduction band and decreasing the electrical resistance of the film.

Response of the films to ethanol gas is quantitatively expressed by sensitivity that calculated using equation 1 [6,7] below:

 $S = [(Re - Ro)/Ro] \times 100 \%$

where, S is the sensitivity of the film, Ro is the electrical resistance of the film in air and Re is the electrical resistance of the film in air containing ethanol gas. Operation temperature was taken from sensitivity data where the sensitivity value is the largest one at lower temperature range. The operation temperature for the thick films is shown in Table 2. From Table 2, it can be seen that the best film is that prepared using screen size of 183 mesh. This film can be operated at relatively low temperature ($120^{\circ}C$) with high sensitivity (65%).



Figure 10. Sample design for ethanol gas sensor

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Figure 11. Electrical resistance as function of temperature of the thick films in air and air containing ethanol gas.

Thickness of film (μm) / Screen Size(mesh)	Operation temperature (°C)	Sensitivity (S) (%)
60/183	120	65
30/225	210	50
20/375	240	55

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4 Conclusion

Thick film ceramics utilizing Fe_2O_3 nano powder derived from yarosite mineral have been fabricated using screen printing method with screen size of 183 mesh, 225 mesh and 375 mesh. The thick films had a semiconductor property. Electrical resistance of the films decreases with the increase of temperature and the increase of the film thickness. The electrical resistance of the films in ethanol gas is lower than that of the films in air indicating that the films had potentiality for ethanol gas sensor.

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References

- C. Doroftei, E. Rezlescu, N. Rezlescu, P. D. Popa, Rom. Journ. Phys., Vol. 51, Nos. 5–6, pp. 631–640, 2006.
- [2] C. Liewhiran, S. Phanichphant, Sensors 7, pp.1159-1184, 2007.

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- [3] G.S. Guo, Y. Wang, W. Ren, F. Gu, H.Y.Guo, Solid State Phenomena Vols 121-123, pp 61-64,2007.
- [4] B. Cela, D. A. de Macedo, G. L. de Souza, R. M. do Nascimento, A. E. Martinelli, C. A. Paskocimas, J. New Mater. Electrochem. Sys. 12,109-113 (2009).
- [5] K. S. Rathore, D. Patidar, Y. Janu, N. S. Saxena, K. Sharma, T. P. Sharma, *Chalcogenide Lett.* 5 (6), 105-110 (2008).
- [6] D. R. Patil, L.A. Patil, D.P.Amalnerkar, Bull. Mater. Sci. Vol.30, No.6, pp.553-559 (2007).
- [7] A. Reungchaiwat, T.Wongchanapiboon, S. Liaruangrath, S. Phanichphant, Sensors, 7, pp. 201-213 (2007).

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