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Optical Absorption Spectra of Mn^{2+} in of $(C_6H_5CH_2CH_2NH_3)_2$-$MnCl_4$ and $(NH_2CH_2CH_2NH_2)_2$-$MnCl_4$ Hybrid Compounds

Authors: M.A. Saleh, Agustinus Agung Nugroho, K.K. Dewi, A.R. Supandi, Djulia Onggo, H. Kuhn, P.H.M. van Loosdrecht

Abstract: The flexibility in replacing the organic part of the organic-inorganic hybrid material has become an interesting material parameter ...more

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Effect of Sintering Temperature on Crystal Structure and Surface Morphology of $NdFeO_3$ Oxide Alloy Materials Prepared by Solid Reaction Method

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Abstract. This research to study the effect of sintering temperature on the crystalline structure and surface morphology of $NdFeO_3$ oxide alloy materials. $NdFeO_3$ was synthesized by solid-state reaction method with the mixing of 99.9% Nd_2O_3 and 99.9% Fe_2O_3 as precursors. Three samples with the different process were made in this experiment. The 1st (#1) and 2nd (#2) samples were sintered for 84 hours at 950°C and 600°C. Calcination process was done at 950°C for 50 hours. The 3rd (#3) sample was sintered for 84 hours at 600°C without calcination process. The samples were characterized by using SEM (Scanning Electron Microscopy) and XRD (X-Ray Diffraction). Based on the SEM characterization result, it was obtained that the sintering temperature influence on surface morphology of $NdFeO_3$ grain size. The XRD analyze was obtained FWHM (Full Width at Half Maximum) value of sample #1, #2 and #3 are 0.11°, 0.10°, and 0.31°, respectively. The value of FWHM was associated with the peak at 2Θ of 32.53° for all sample; it is indicated of *hkl* (121). Further calculation based on crystallography data was carried out by Rietveld method with rietica software, and the best quality will be applied as a gas sensor materials.

1. Introduction

Combustion-related emission gases usually include CO_2, CO, hydrocarbons (HCs), volatile organic compounds (VOCs), NO_x and O_2 [1]. According to Euro-VI, the limit values for CO, NO_x, and HCs are 1, 0.06 and 0.1 g km−1, respectively. The exhaust gases are the relatively corrosive environment, for example, the vehicle exhaust gases CO, HCs and NO_x have concentrations up to a level of 10, 0.2 and 0.1 vol.%, respectively [2]. They are highly active and poisonous gases. For this reason, control and measurement of the concentrations of these gases are very important in reducing environmental pollution.

The nano-perovskite oxides with the structure of *ABO*3 (*A*: La, Nd, Sm, and Gd; *B*: Fe, Co and Ni; and O: oxygen) have a high catalytic activity and high sensitivity with CO and HCs. Their applications in gas sensors were studied especially for the properties in electrical and response for CO gas [3]. The rare-earth orthoferrites, having perovskite structure of $YFeO_3$ have been investigated in a wide variety of applications such as solid-oxide fuel cells [1], gas sensors [2,4], photo-catalysis and vehicle catalytic converters [5-7]. $NdFeO_3$ (NFO) is a perovskite transition metal oxide with an orthorhombic structure and space group of Pbnm possesses insulator character at room temperature [8].

$NdFeO_3$ have a high sensitivity on CO, H_2S, and ethanol [9]. In this study, $NdFeO_3$ sample was synthesized by solid-state reaction method. In this paper, we reported the result of the effect of sintering temperature on the crystalline structure and surface morphology of $Nd_{1+x}FeO_3$.

2. Experimental Method

$Nd_{1+x}FeO_3$ oxide alloy was synthesized using raw material powder of Nd_2O_3 (99.9%) and Fe_2O_3 (99.9%) by solid-state reaction method. Preparation of three samples $NdFeO_3$ material used same molar ratio which is x = 0. The initial calculation of raw material mass used Proust law with the chemical formula as shown in equation (1):

$$Nd_2O_3 + Fe_2O_3 \rightarrow 2NdFeO_3 \quad (1)$$

The mixing of powder with raw material was crushed by mortar and pastel for 3 hours to maximize the reaction by extending the contact between the surface of reactants and reagents be mixed homogeneously. The #1 sample was calcined at 950˚C for 50 hours and then crushed again for 5 hours. The samples were pelleted and sintered at 950˚C for 84 hours. The #2 sample was calcined at 950˚C for 50 hours and then crushed again for 5 hours. The samples were pelleted and sintered at 600˚C for 84 hours. The #3 sample was sintered at 600˚C for 84 hours without calcination process. All the samples were synthesized and then characterized using XRD and SEM to identify and analyze the crystalline structure and surface morphology.

3. Results and discussion

The XRD spectra are shown in figure 1. The spectrum shows the increase of the peak intensity and widens of the width peaks when the sintering temperature is added. Figure 1 shown the highest intensity variation of the sample #1 at 2Θ of 32.53˚ which are related with the intensity of 984.56 counts, followed by sample #2 at 2Θ of 32.53˚ related with the intensity of 216.84 counts, and sample #3 at 2Θ of 32.53˚related with the intensity of 53.89 counts, respectively. All of the samples showed (*hkl*) position at (121). For sample #3, (*hkl*) position at (121) is not dominant. This result indicates that the raw materials powder has well reacted during the preparation process to form of $NdFeO_3$ alloy, as was reported elsewhere [8].

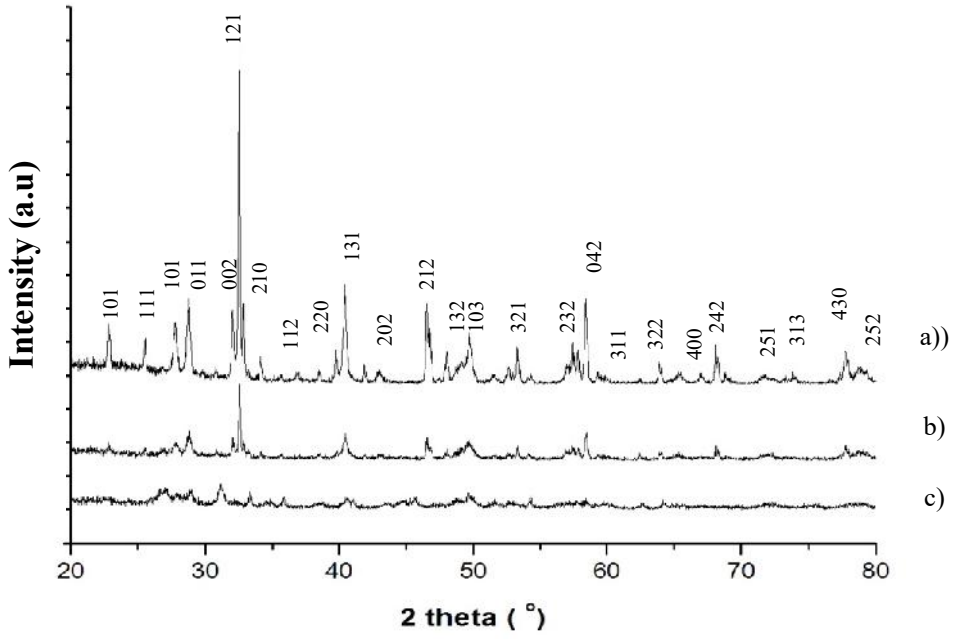

Figure 1. XRD spectra of sample $NdFeO_3$ alloy a) sample #1, b) sample #2, and c) sample #3 was prepared using solid-state reaction method.

The XRD characterization result obtained that the sintering temperature influence of the FWHM value. The FWHM value is 0.11˚, 0.10˚ and 0.31˚ for sample #1, sample #2 and sample #3, respectively. The result we know that for adding sintering temperature is not really influenced FWHM, it is has shown the FWHM of sample #1 is wider than sample #2.

The highest intensity and FWHM value obtained the best quality of the crystalline structure is for sample #1 this is related with the intensity of 984.56 counts and 0.11˚. This result is similar with reference had reported[1,5,6,10-15], although the sample prepared using a different method.

Phases analysis using *Match!* the software showed that there are four phases that contain in each sample: $NdFeO_3$, $Nd(OH)_3$, Fe_2O_3 and Nd_2O_3

Table 1. Phase analysis of the samples $Nd_{1+x}FeO_3$.

Sample	$NdFeO_3$ (%)	Nd_2O_3 (%)	Fe_2O_3 (%)	$Nd(OH)_3$ (%)
#1	74	2	14	10
#2	72	11	8	9
#3	55	7	38	-

The data of phase composition as described in table 1 indicate that each of sample was containing a few percents of $Nd(OH)_3$ and there are still formed Nd_2O_3 and Fe_2O_3 phase. This means that the temperature was used to synthesis $NdFeO_3$ alloy should be higher than 950˚C or another way with increased the heating time to get perfectly reaction that occurs and lattice -OH was missing.

The morphology, structure and particle size of samples $Nd_{1+x}FeO_3$ as a variation of sintering time were investigated by SEM. Figure 2 shows the SEM micrograph of the samples.

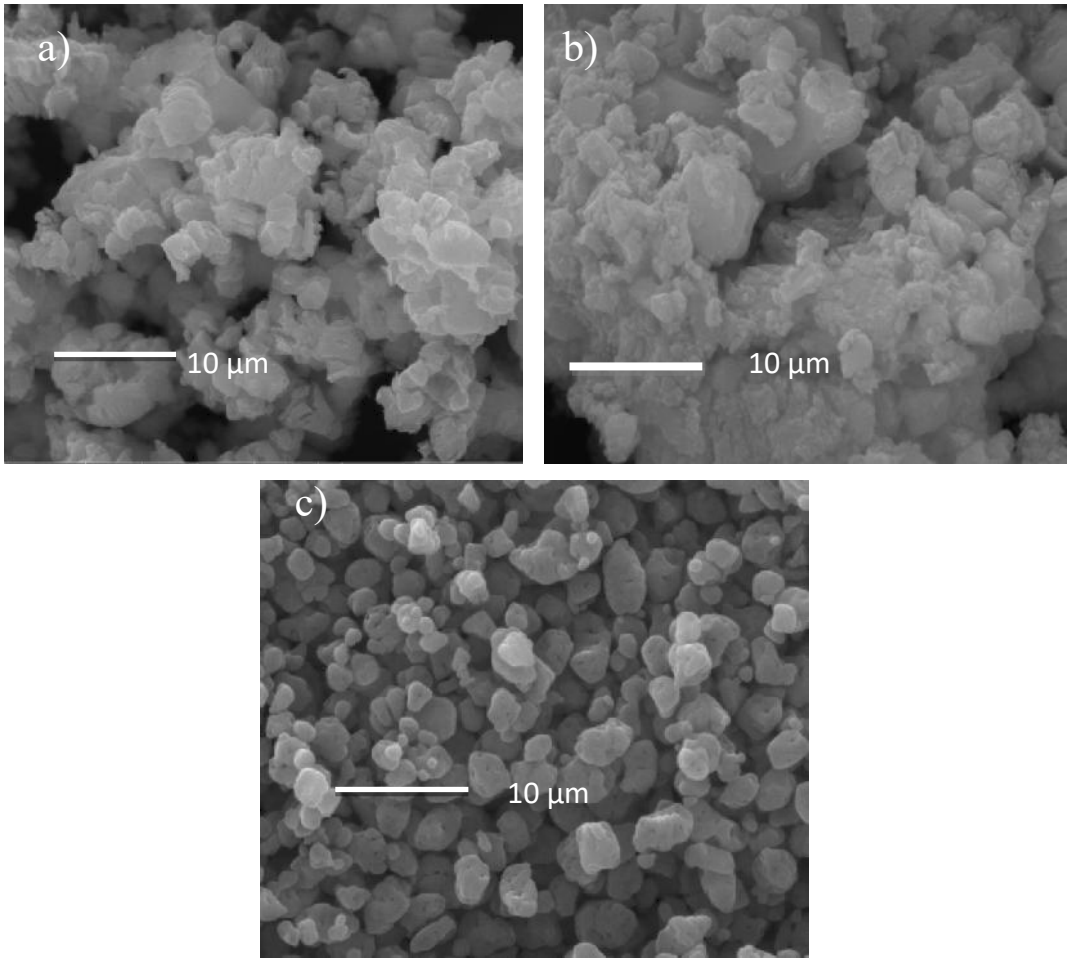

Figure 2. SEM image of $NdFeO_3$ alloy a) sample #1, b) sample #2 and c) sample #3, respectively.

Figure 2 shown SEM image of $NdFeO_3$ alloy was prepared with different parameter process. As shown in the figure, 2a) that grain size of the $NdFeO_3$ alloy formed well connected and there is did not observe grain boundaries. This data indicated that the result of phase composition was obtained is confirmed. On the other hand, based on figure 2c) shows that many of grain size shape were observe and this is caused by appearing of grain boundaries. Its means the raw material is not reacted perfectly as also confirmed from the phase composition of the result as sample #3.

Determination of crystalline size can be calculated using the Scherrer equation. Based on the calculation was obtained grain size of the samples #1, #2, and #3 are 147.83 ± 0.01nm, 162.69 ± 0.01nm and 52.94 ± 0.01nm, respectively. Similar results were obtained by Khorasani-Motlagh M. et al. that synthesis nanocrystals $NdFeO_3$ materials with has good porosity and the crystalline size ranging from 28.82 nm and 200.71 nm [7]. These results indicate well confirmation between XRD analysis and surface morphology of the SEM image. Therefore we confirmed that the sintering temperature also influences the grain size and grain boundary of the sample was prepared by solid-state reaction [6,9,16].

4. Conclusion

$NdFeO_3$ alloy has been prepared by solid-state reaction method. There is well confirmation between XRD analysis and SEM image. These results indicate that the sintering temperature influences the diffraction intensity, FWHM value, and grain size. Dominated of *hkl* 121 phase of $NdFeO_3$ alloy prepared using solid state reaction are useful for further application as a gas sensor.

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