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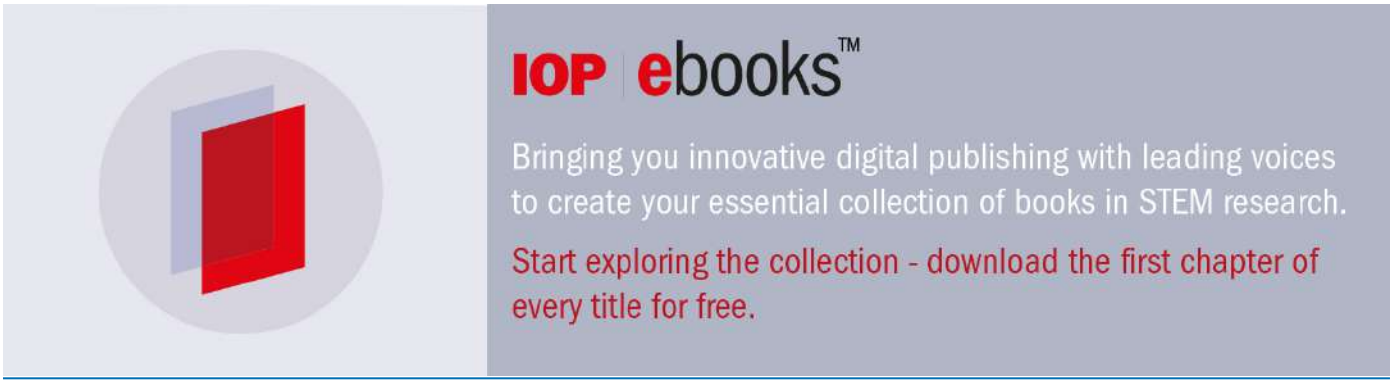

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Influence of High Sintering Temperature Variation on Crystal Structure and Morphology of $Nd_{1.2}FeO_3$ Oxide Alloy Material by Solid-State Reaction Method

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Abstract. $Nd_{1.2}FeO_3$ powders type perovskite structure was prepared by a solid-state reaction method. This research has been conducted with the recurring heating process at high temperature. The raw material consisted of Nd_2O_3 (99.99 %) and Fe_2O_3 (99.99 %) which characterized by XRD to confirm the phase and using SEM to identify the morphology structure of the sample. Result characterized by XRD confirms the phase of $NdFeO_3$ and Nd_2O_3 with the formation of $NdFeO_3$ having the orthorhombic structure (perovskite type). The value of FWHM and the average crystal size of $NdFeO_3$ was obtained for each sample is 0.20º and 409 nm. While SEM studies showed the surface morphology of $Nd_{1.2}FeO_3$ has homogeneous granules with grain size estimates is 0.2 µm. These results indicate that sample $Nd_{1.2}FeO_3$ was a good candidate for gas sensor materials.

Keywords. Crystal structure, morphology, sintering, $NdFeO_3$ oxide alloy, and solid state method.

1. Introduction

Many researchs have been conducted on oxide compounds to be used as a gas sensor, one of them is to use a perovskite oxide that is $NdFeO_3$ synthesized by various methods or techniques [1–5]. $NdFeO_3$ known to have type orthorhombic [1, 3–6]. The nano-perovskite oxides ABO_3 (*A*: La, Nd, Sm, and Gd; *B*: Fe, Co and Ni; and O: oxygen) have high catalytic activities and high sensitivity for gas sensor material. The $NdFeO_3$ is mainly using in gas sensing and catalysis application [2, 3, 6]. Research on $NdFeO_3$ by sol-gel citrate method obtained perovskite-type $NdFeO_3$ can be used as an H_2S gas sensor and catalytic CO gas sensor in exhaust gas environments [3, 6]. Various synthesizing techniques have been used for synthesis $NdFeO_3$ alloy oxide it such as by hydrothermal method [7], combustion [8, 9], sol-gel [10], precipitation method [11], solid-state reaction method [12] and sonication assisted precipitation [13].

The solid-state reaction is one of the oldest synthesis routes for the preparation of perovskites [12]. An advantage of this method is a cheap, simple and fast method for the synthesis perovskite. Also, the product of the reaction has high purity and good crystallinity. The properties of the perovskite materials are closely related to either the preparation or sintering conditions. In the ceramics, the sintering process is essential that effect on microstructure, grain growth, and densification [14].

In this paper, we present perovskite oxide $Nd_{1.2}FeO_3$-based by a solid state method with varying the sintering temperature using heat treatment process. Then the crystal structure and morphology of $Nd_{1.2}FeO_3$ has been characterized by X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM).

2. Materials and methods

The powders oxide $Nd_{1.2}FeO_3$ were prepared by the solid-state reaction method. Raw materials of Nd_2O_3 (Strem Chemicals, 99.99 %) and Fe_2O_3 (Sigma-Aldrich, 99.99 %) were mixed together based on a stoichiometric calculation using a molar ratio of x = 0.2 [15] to get an oxide alloy $Nd_{1.2}FeO_3$. Then powders were grinded for 3 h and calcined using furnace at 700 °C for 6 h. Sample powders are resulting calcination then were grinded back for 5 h to get a homogeneous mixture and sintered at 950 °C for 6 h.

The synthesis process and the heating are then repeated to obtain a better sample homogeneity [16]. Then mix powders produced were grinded for 3 h and calcined at 950 °C for 6 h. Then the result of the calcined sample was grinded back for 5 h to maximize reaction and to increase the homogeneity and sintered as a variation of high temperatures 1000 °C, 1050 °C, and 1100 °C for 6 h, respectively. Finally, the samples were annealed at 450 °C for 1 h. Powders oxide $Nd_{1.2}FeO_3$ were characterized by XRD type Rigaku MiniFlex II 2θ = 10° to 70° (Cu Kα, λ = 0.154 nm) and SEM-EDS type Tescan Vega3SB to analyzed the phase composition and to confirm morphology and elemental structure.

3. Results and discussion

X-ray diffraction (XRD) pattern of samples $Nd_{1.2}FeO_3$ at high temperatures 1000 °C, 1050 °C, and 1100 °C shown in Figure 1, respectively. According to the result of synthesis $NdFeO_3$ powder obtained diffraction peaks form a single phase with a perovskite structure [4, 17]. In Figure 1 shows the formation phase of $NdFeO_3$ and Nd_2O_3. Formation of phase Nd_2O_3 produces another peak that regarded as an impurity phase and can reduce the sensitivity of the material as a gas sensor. Phases analysis using *Match!* software shows that dominant phase of $NdFeO_3$ is having the orthorhombic structure (perovskite type) with Pnma space group [18].

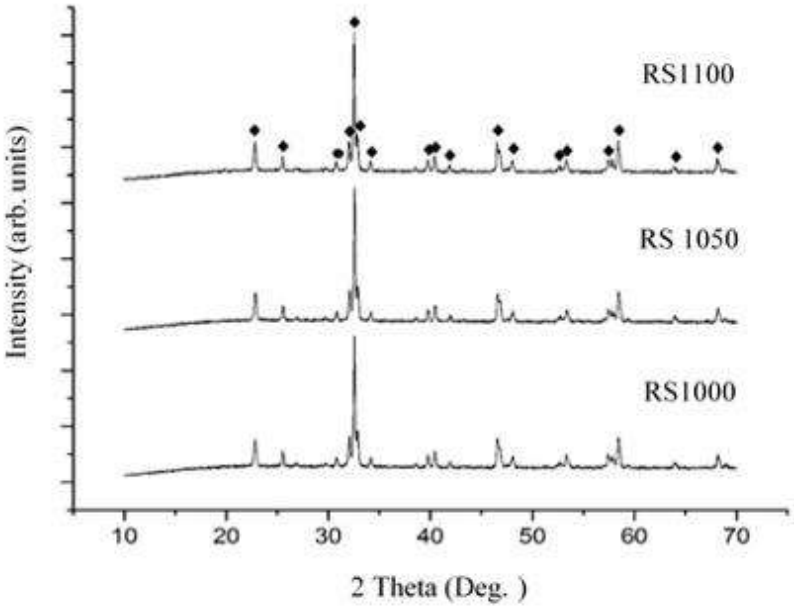

Figure 1. XRD patterns of $Nd_{1.2}FeO_3$ as variation of different termperatures (♦= $NdFeO_3$, ●= Nd_2O_3).

Table 1. Data of positions (2θ), intensities and FWHM values of $Nd_{1.2}FeO_3$ phases

Sample	Identification phase	2θ (º)	Intensity (Counts)	FWHM (º)
RS1000	$NdFeO_3$	32.54	13053.33	0.20±0.02
RS1050	$NdFeO_3$	32.56	13260.00	0.20±0.02
RS1100	$NdFeO_3$	32.52	13810.00	0.20±0.02

It can be seen from Table 1, FWHM values of $NdFeO_3$ phase same for all three samples at the dominant peak is *hkl* (121) with the peak position 2θ = 32.5°. Synthesis of $NdFeO_3$ has also been done by Jada Shanker *et al.* with temperature 900 °C [5], and Yabin Wang *et al.* with temperature 1000 °C [4] which get similar results that phase $NdFeO_3$ exist at 2θ = 32.56° with *hkl* (121). FWHM value stated of homogeneity between atoms in a crystal which the smaller the FWHM value means the lattice more homogeneous or has good crystallinity, which means that the level of material quality is also getting better [4]. The crystal size can be calculated using *Debye-Scherrer* equation (1):

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

where λ is the wavelength of X-ray (0.15405 nm for Cu Kα), β is FWHM value at *hkl* (121), and θ is diffraction angle. Based on the calculation, the value of the crystal size of $Nd_{1.2}FeO_3$ which synthesized was 409.37 nm, 409.39 nm and 409.35 nm, respectively.

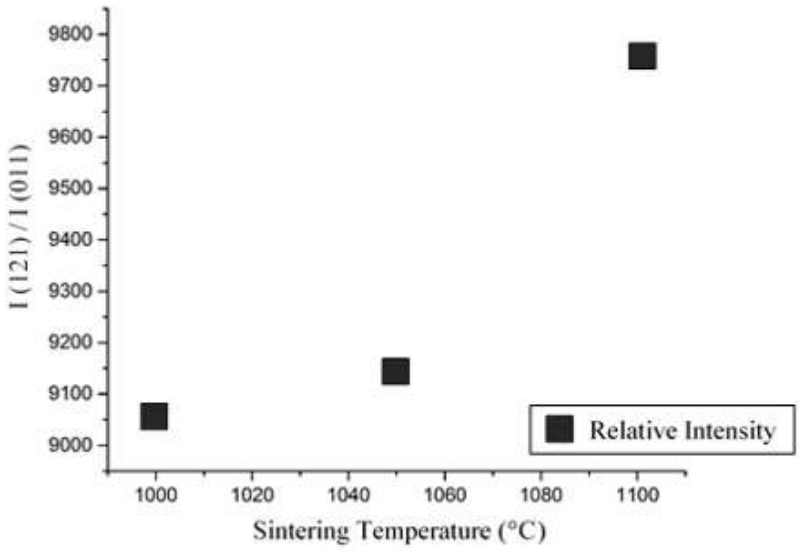

Figure 2. Relative intensity $I(121)/I(011)$ of $Nd_{1.2}FeO_3$ powders oxide material as a function of sintering temperatures.

Figure 2 shows diffraction intensity curve between the dominant of peak intensity (121) with a peak intensity of (011) for each variation of sintering temperature. In this study, we found that a significant change of peak intensity of (011) as a comparison of (121) which is indicating that the intensity of Nd_2O_3 phase decreases as the temperature of sintering increases.

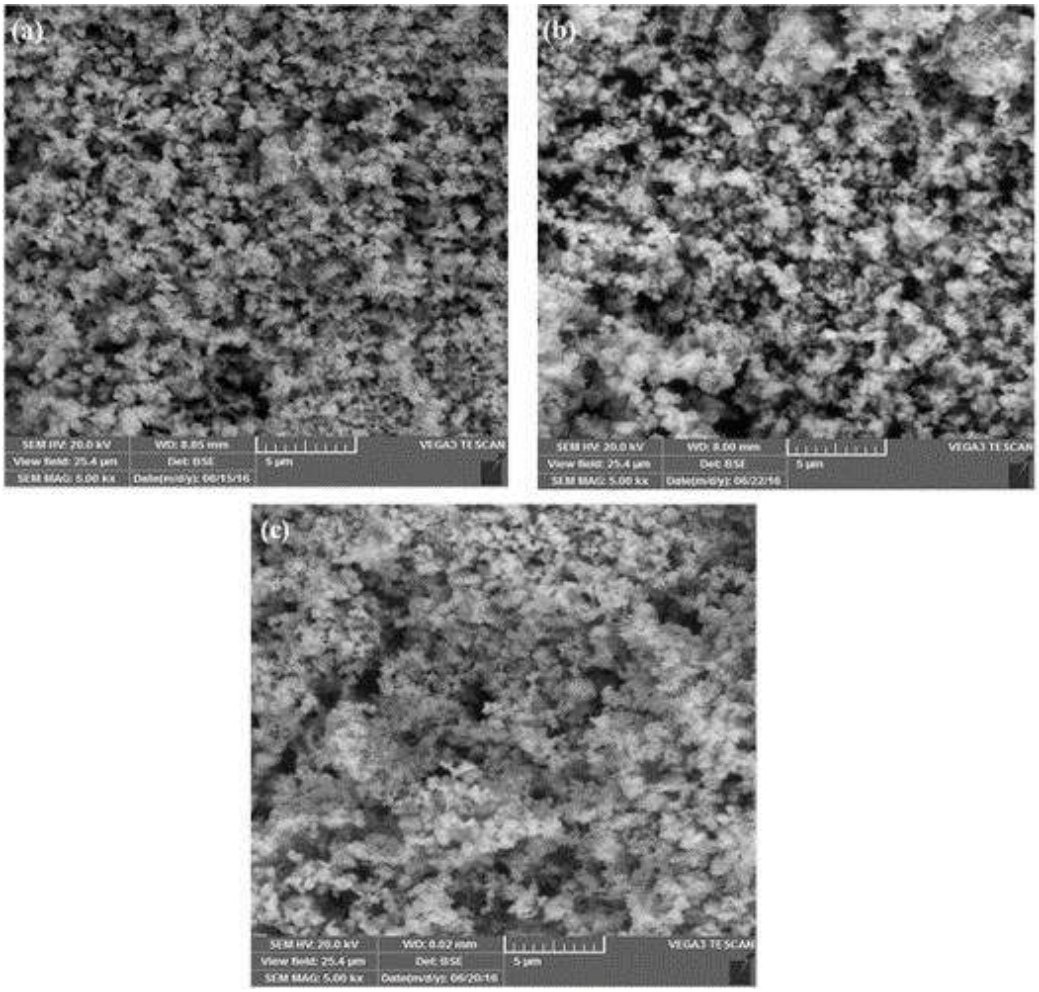

Figure 3. SEM images of sample $Nd_{1.2}FeO_3$ as a variation of sintering temperature a) RS1000, b) RS1050, and c) RS1100, respectively.

Table 2. Data composition element of $Nd_{1.2}FeO_3$ samples using EDS

Compound	Norm. C [wt%]			Error (3 Sigma) [wt%]		
Norm.	RS1000	RS1050	RS1100	RS1000	RS1050	RS1100
Oxygen	16.94	16.50	16.41	5.91	5.83	6.90
Sodium	1.32	-	-	0.38	-	-
Magnesium	0.27	-	-	0.15	-	-
Aluminium	0.25	-	-	0.14	-	-
Potassium	0.04	-	-	0.09	-	-
Titanium	0.11	-	-	0.10	-	-
Iron	21.11	21.60	19.87	1.63	1.70	1.77
Copper	0.85	0.81	0.38	1.22	0.22	0.19
Neodymium	58.99	60.94	63.15	4.26	4.52	5.23
Silicon	0.12	0.15	0.18	0.11	0.11	0.13

The morphology, crystal structure and particle size of the sample were investigated by SEM. Microstructures of polycrystalline $Nd_{1.2}FeO_3$ sintered at various temperatures are shown in Figure 3. SEM results of all samples in Figure 3, it is generally assumed that the powders $Nd_{1.2}FeO_3$ oxide alloy material have a high homogeneity level with formation small uniform granules which an estimated grain size about of 0.2 μm and the color is almost evenly gray. This powder has a very high porosity,

and this is an advantage for improving the gas-sensing characters, as has reported by Niu Xinshu *et al.* [2] and Ho *et al.* [3].

Table 2 shows the elemental composition of samples $Nd_{1.2}FeO_3$, were RS1000 contain Nd (58.99 wt%) and Fe (21.11 wt%), RS1050 contain Nd (60.94 wt%) and Fe (21.60 wt%), and RS1100 contain Nd (63.15 wt%) and Fe (19.87 wt%), which also contain a minor phase as shown in Table 2. That minor phase as indication due to the sample holder preparation process and no significant effect on the constituent elements of each sample.

4. Conclusions

The perovskite oxide $Nd_{1.2}FeO_3$ powders have been prepared by a solid-state method with the recurring heating process at high temperatures of 1000 °C, 1050 °C, and 1100 °C, respectively. The result of X-Ray diffraction analysis showed the phase of $NdFeO_3$ and Nd_2O_3 which phase $NdFeO_3$ is an orthorhombic structure with Pnma space group. The results indicate that $Nd_{1.2}FeO_3$ analyzed at high-temperature variation given a relatively stable quality, it can be seen the same FWHM value is 0.20° with an estimated crystalline size about of 409 nm. While SEM studies showed, the surface morphology of $Nd_{1.2}FeO_3$ has homogeneous granules and high porosity with an estimated grain size of 0.2 μm. The results indicate that $Nd_{1.2}FeO_3$ is a good candidate for gas sensor materials as has reported elsewhere.

Acknowledgments

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