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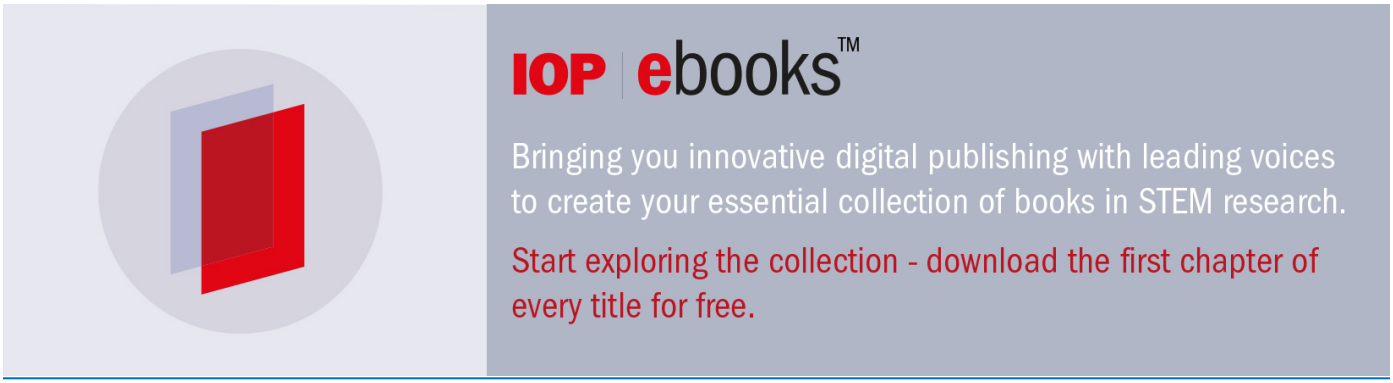

Influence of Annealing Time Variation on Crystal Structure and Morphology of Oxide Material $Nd_{1.2}FeO_3$ by Solid-State Reaction Method

E. H. Sujiono[1, *)], A.C. M. Said[1], M. Y. Dahlan[1], R. A. Imran[1], and S. Samnur[1]

[1]Laboratory of Materials Physics, Department of Physics, *Universitas Negeri Makassar,* Makassar 90224, Indonesia.

E-mail: e.h.sujiono@unm.ac.id

Abstract. $NdFeO_3$ is one of the oxide material can be detected various gases, like S/O_2, CO, H_2S, etc. In this research, $Nd_{1.2}FeO_3$ as oxide material have been synthesized by solid-state reaction with a variation of annealing time. Characterized by XRD shows that the samples have form crystal perovskite structure with dominant phase and peak intensity correspond to *hkl* (121). FWHM value for the dominant peak was 0.22°. The crystallite of the samples was determined using Debye Scherer formula were 393.08, 393.10, and 393.10 nm, respectively. While the SEM characterized showed the morphology of the samples was homogenous with grain size estimates of 0.2μm. These results indicate the variation of annealing time 1, 2, and 3 hours did not significantly affect the crystallinity and morphology of $Nd_{1.2}FeO_3$ oxide material.

Keywords. Annealing time, crystallinity, morphology, $Nd_{1.2}FeO_3$ oxide material, and solid-state reaction.

1. Introduction

Currently in the modern chemical industry mixed metal oxide including perovskite oxide structure with general structure ABO_3 where A was a rare-earth element and B was 3D transition metal had been used remain prominent [1]. Iron was metal in the first transition series that has an orthorhombic structure with perovskite-type that was a subject of extensive investigated [2–4]. A number of research shown their utility in a wide range of applications such as a catalytic gas sensor, magnetic material, pigment material, fuel cells, gas sensor, etc. [2–6]. As a gas sensor, $NdFeO_3$ was very efficient for H_2S, CO and LPG detection [6–8].

Material oxide could be synthesized in several ways, such as ceramic method, chemical precipitation process, float zone technique, sol-gel and solid-state reaction [3,8–12]. The solid-state reaction was one of the most conventional methods, which was an easy and inexpensive method to a synthesized oxide material. The method was mixed different metal oxide alloys at high temperatures [13]. In this research, $Nd_{1.2}FeO_3$ oxide material was synthesized by solid-state reaction. Characteristic of the crystalline structure of oxide material was studied using X-ray diffraction (XRD) and the morphology using Scanning Electron Microscopy (SEM). The full width at half maximum (FWHM) is one effective method to confirm the

crystal quality and the morphology of the material. The crystal quality has category higher due to the smaller of FWHM value and the crystal size in dimension to nanometer [14].

In this paper, presented influence of annealing time (t) as a variation of 1 h, 2 h, 3 h on crystal structure and morphology of $Nd_{1.2}FeO_3$ synthesized by solid-state reaction method. Variation of annealing time has an effective treatment for removing structural grain boundaries of ionic defects [15]. It is an indication that annealing process would increase the crystal quality of the material. The composition of $Nd_{1.2}FeO_3$ used has been reported elsewhere [16]. Therefore, this paper would be a focus on appearing results of variation annealing time on crystal quality and morphology.

2. Materials and methods

The $Nd_{1.2}FeO_3$ have been synthesized by solid stated reaction method with repeated heating treatment. The synthesized process was started by the mixed stoichiometric powder of Nd_2O_3 99.99 % (*Strem Chemicals*) and Fe_2O_3 99.99 % (*Sigma-Aldrich*) and grinded them for 3 h [16]. The samples were calcinated at 700 °C for 6 h. After that sample were grinded for 5 h and sintered at 950 °C for 6 h. This process was called the first heat treatment. Then, the sample was grinded for 3 h and calcined at 950 °C for 6 h. The last, the sample was grinded for 5 h and continued using *in situ* annealed at 450 °C for 1 h, 2 h, and 3 h, respectively. This process was called the second heat treatment.

The synthesized powder samples of $Nd_{1.2}FeO_3$ were characterized by using x-ray diffraction (Rigaku Miniflex II CuKα, λ = 0.154 mm) and Scanning Electron Microscopy (Tescam Vega-SB) for analyzed crystal structure and the morphology of the samples.

3. Results and discussion

In this research, the samples have characterized by X-ray diffraction (XRD). The intensity was observed over 2θ range 10° to 70°. The XRD pattern of the $Nd_{1.2}FeO_3$ oxide material is shown in Figure 1. The pattern has formed a crystal with the orthorhombic phase of perovskite-type (JCPDS File no 25 – 1149) with the highest peak at 2θ = 32.5° and correspond to *hkl* (121) plane. This result has similar as reported by Satyendra Singh et al., with annealing temperature at 450 °C for 2 h [17].

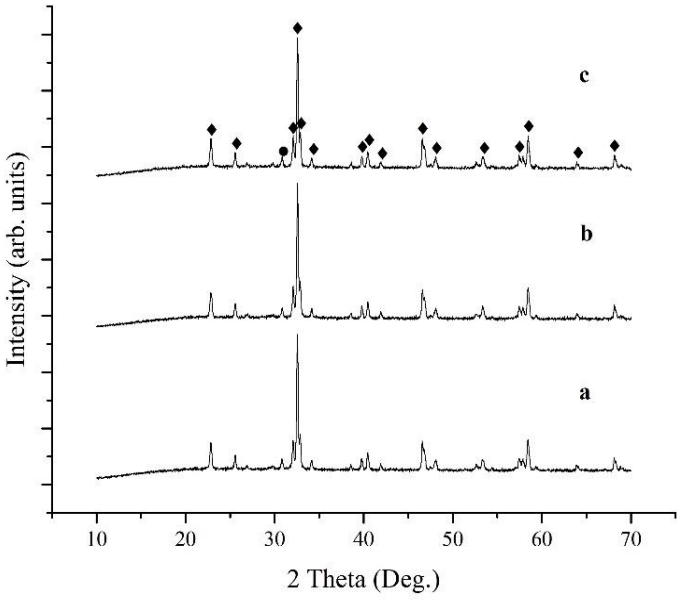

Figure 1. XRD pattern of $Nd_{1.2}FeO_3$ as variation of annealing time a = 1 h, b = 2 h, and c = 3 h, respectively (♦= $NdFeO_3$, ●= Nd_2O_3).

In this research, we also found that the highest peak intensity is corresponding to *hkl* (121). The peak was a very sensitive peak as a gas sensor, reported by Niu Xinshuet et al. The *hkl* (121) has high sensitivity, excellent selectivity, and quick response and recovery behavior to H_2S [6]. The (121) peak also suitable for detecting the CO gas as reported by Truong Giang Ho [2]. Based on this result and references indicate that $Nd_{1.2}FeO_3$ was a good material as a gas sensor.

The Rietveld analysis using Rietica software had refinement results of the lattice parameters for each sample. Lattice parameters for samples with annealed time varied for 1 h, 2 h, and 3 h has value is a = 5.58 Å, b = 7.76 Å, c = 5.45 Å, this similar as reported [10]. The average *Goodness of Fit* (GoF) for samples was 0.91%, profile (Rp) 4.63 %, Weighted Profile (Rwp) 6.09 %, and Expected (Rexp) 6.39 %, this data indicates a good agreement within the experiment result and database calculation as reported elsewhere [18].

Phase analysis using Match software obtaining two phases that contain each $Nd_{1.2}FeO_3$ oxide materials as a variation of annealing time. There is was observed in Figure 1, peak with attribute circle correspond to Nd_2O_3 and diamond to $Nd_{1.2}FeO_3$. The samples suspected were not completely reacted and did not form a single phase of $NdFeO_3$ oxide material. Increasing intensity of peak $Nd_{1.2}FeO_3$ (121) would be accompanied by the decreased intensity of peak Nd_2O_3 (011) as confirmed in Figure 2. It was mean that Nd_2O_3 as a raw material was not perfectly reacted Fe_2O_3 to form $NdFeO_3$ sintered at 950 °C and annealed time varied at 1 h, 2 h, and 3 h. The suggestion it was needed a higher temperature and longer time to heat treatment process to form perfect $Nd_{1.2}FeO_3$ phase. Figure 2 was shown sample was annealed time to 2 h has higher ratio intensity between $Nd_{1.2}FeO_3$ (121) and Nd_2O_3 (011).

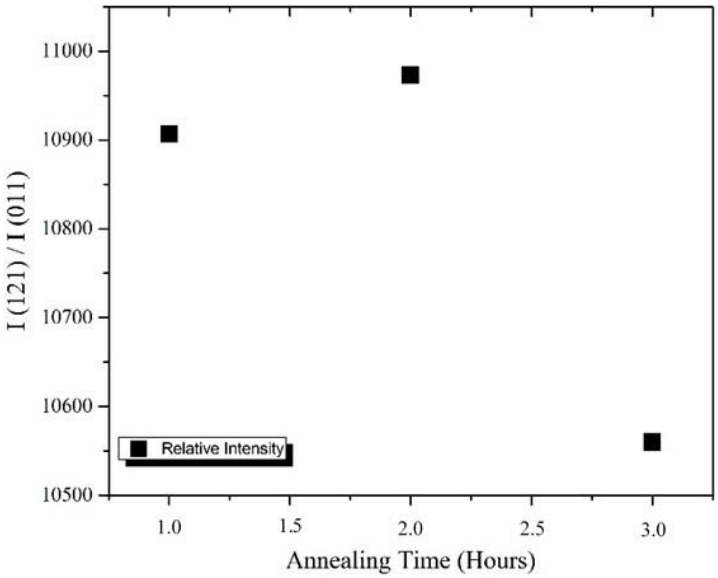

Figure 2. Curve comparison relative intensity of $Nd_{1.2}FeO_3$ (121) and Nd_2O_3 (011) for each variation of annealing time

The crystallite size of oxide material was calculated by Debye Scherer's formula, which is given as:

$$D = \frac{0.94\lambda}{\beta_{hkl}\cos\theta} \quad (1)$$

where D is the crystallite size, β_{hkl} is the full width at half maximum (FWHM) intensity value, θ is the diffraction angle, and λ represents the x-ray wavelength. The crystallite size of $Nd_{1.2}FeO_3$ was shown in

Table 1. Based on the result as was described in Table 1, phase and crystallite size have similar than previous research [16].

Table 1. Result of XRD analysis for $Nd_{1.2}FeO_3$oxide material

Annealing time (h)	2θ (°)	FWHM (°)	Crystallite Size (nm)
1	32.56	0.22	|393.08 ± 0.02|
2	32.58	0.22	|393.10 ± 0.02|
3	32.58	0.22	|393.10 ± 0.02|

The surface morphology, structure and particle size of the samples were investigated by SEM. Figure 3 was shown the SEM micrograph of the samples at 5000 magnification. SEM micrograph showed that morphology of samples was homogeneous grain orientation and grain size with an estimated size was 0.2 μm. It can be seen, a variation of annealing time as a parameter process has obtained nanostructure with uniform in both morphology and particle size. This result confirms that annealing time at 2 h has more homogeneous morphology [17].

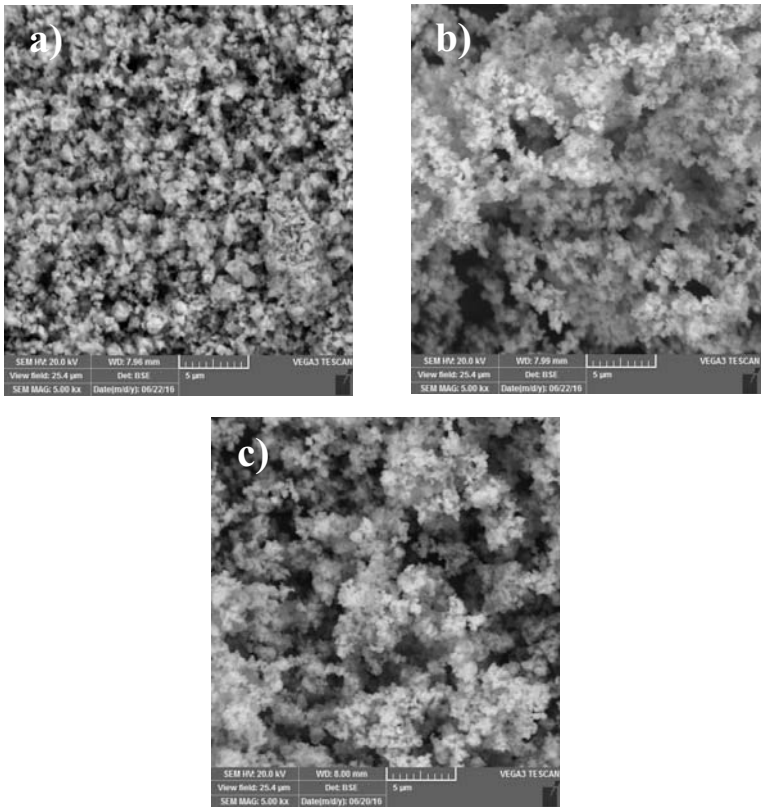

Figure 3. SEM micrograph of $Nd_{1.2}FeO_3$ as variation of annealing time t of (a) 1 h, (b) 2 h, and (c) 3 h, respectively.

EDS analysis results are given in Table 2. The results showed the existence of iron and neodymium as a dominant element. In addition, it can be seen the percentage value the presence of another minor oxide in each sample due to the sample holder.

Table 2. EDS analysis of $Nd_{1.2}FeO_3$ oxide material as variation of annealing time

Compound Norm.	Comp. C [wt%]		
	t = 1 h	t = 2 h	t = 3 h
Nd	71.44	67.95	70.77
Fe	26.86	26.36	26.90

Based on the XRD and SEM analysis has been describing above, the variation of annealing time did not significantly affect on crystalline quality and morphology of $Nd_{1.2}FeO_3$ oxide material. Annealing was not a process to form a new phase, but to maintain the oxygen ion of an oxide material. Therefore annealing treatment not affect significantly the crystal structure and sample morphology, but will affect the electrical properties of the material. This phenomenon was similar to the case of $YBa_2Cu_3O_{7-\delta}$ oxide material which is annealing did not affect the crystal structure and morphology of material. Annealing affects the conductivity of the sample as corresponding to oxygen composition [14, 19, 20]. This also occurred in case of TiO_2 material that annealing variation affects the resistivity of the material [21].

4. Conclusions

$Nd_{1.2}FeO_3$ oxide material has been synthesized by solid-state reaction method with a variation of annealing time. XRD result shown the oxide has a crystal structure with the orthorhombic phase of perovskite-type with the highest peak of *hkl* (121) at 2θ is 32.5º. There were $Nd_{1.2}FeO_3$ and Nd_2O_3 phases contain in each sample. The calculation using Debye Scherer's formula given crystallite size is 393 nm with FWHM 0.22°. SEM micrograph of the samples shown morphology was uniform grain size around 0.2 μm. Based on XRD and SEM results, a sample with annealing time for 2 h has ratio intensity of *hkl* (121) more dominant as an indication of the best sample in this research.

Acknowledgements

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