

Table of contents

Volume 367

2018

◀ Previous issue Next issue ▶

The 5th International Conference on Advanced Materials Sciences and Technology (ICAMST 2017) 19–20 September 2017, Makassar, Indonesia

Accepted papers received: 16 May 2018

Published online: 12 June 2018

Open all abstracts

Preface

OPEN ACCESS 011001

The 5th International Conference on Advanced Materials Sciences and Technology (ICAMST 2017)

+ Open abstract  View article  PDF

OPEN ACCESS 011002

The List of Committee ICAMST 2017

+ Open abstract  View article  PDF

OPEN ACCESS 011003

List of Participant ICAMST 2017

+ Open abstract  View article  PDF

OPEN ACCESS 011004

Photographs


+ Open abstract  View article  PDF

OPEN ACCESS 011005

Peer review statement

+ Open abstract  View article  PDF

Papers

OPEN ACCESS 012001
This site uses cookies. By continuing to use this site you agree to our use of cookies. To find out more, see our Privacy and Cookies policy. 

OPEN ACCESS

012037

Influence of Annealing Time Variation on Crystal Structure and Morphology of Oxide Material $\text{Nd}_{1,2}\text{FeO}_3$ by Solid-State Reaction Method

E. H. Sujiono, A.C. M. Said, M. Y. Dahlan, R. A. Imran and S. Samnur

[+ Open abstract](#) [View article](#) [PDF](#)

OPEN ACCESS

012038

Porous Fe_2O_3 Microspheres as Anode for Lithium-Ion Batteries

L. Noerochim, M. A. T. Indra, H. Purwaningsih and A. Subhan

[+ Open abstract](#) [View article](#) [PDF](#)

OPEN ACCESS

012039

Analysis of Porosity Defects in Aluminum as Part Handle Motor Vehicle Lever Processed by High-pressure Die Casting

L. Anggraini and Sugeng

[+ Open abstract](#) [View article](#) [PDF](#)

OPEN ACCESS

012040

Synthesis and Characterization of Barium-Hexaferrite-Based Nanocomposite on X-Band Microwave

Y. E. Gunanto, M. P. Izaak, S. S. Silaban and W. A. Adi

[+ Open abstract](#) [View article](#) [PDF](#)

OPEN ACCESS

012041

Semiconductor Ceramic $\text{Mn}_{0,5}\text{Fe}_{1,5}\text{O}_3\text{-Fe}_2\text{O}_3$ from Natural Minerals as Ethanol Gas Sensors

H. Aliah, D. G. Syarif, R. N. Iman, A. Sawitri, M Sanjaya WS, M. Nurul Subkhi and P. Pitriana

[+ Open abstract](#) [View article](#) [PDF](#)

OPEN ACCESS

012042

The Effects of Calcination Temperatures on Crystal Structures and Morphologies of $\text{Nd}_{1,2}\text{FeO}_3$ Synthesized by Solid-State Reaction

E. H. Sujiono, M. Y. Dahlan, R. A. Imran, A.C. M. Said and S. Samnur

[+ Open abstract](#) [View article](#) [PDF](#)

OPEN ACCESS

012043

Synthesis and Characterization of Monodisperse Core-shell Lanthanide Upconversion Nanoparticles $\text{NaYF}_4\text{: Yb,Tm/SiO}_2$

R. V. Manurung, G. Wiranto and I. D. P. Hermida

[+ Open abstract](#) [View article](#) [PDF](#)

PAPER • OPEN ACCESS

Influence of Annealing Time Variation on Crystal Structure and Morphology of Oxide Material $\text{Nd}_{1.2}\text{FeO}_3$ by Solid-State Reaction Method

To cite this article: E. H. Sujiono *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **367** 012037

View the [article online](#) for updates and enhancements.

Related content

- [Influence of High Sintering Temperature Variation on Crystal Structure and Morphology of \$\text{Nd}_{1.2}\text{FeO}_3\$ Oxide Alloy Material by Solid-State Reaction Method](#)
E. H. Sujiono, R. A. Imran, M. Y. Dahlan et al.
- [The Effects of Calcination Temperatures on Crystal Structures and Morphologies of \$\text{Nd}_{1.2}\text{FeO}_3\$ Synthesized by Solid-State Reaction](#)
E. H. Sujiono, M. Y. Dahlan, R. A. Imran et al.
- [Effects of Molar Ratios and Sintering Times on Crystal Structures and Surface Morphology of \$\text{Nd}_{1+x}\text{FeO}_3\$ Oxide Alloy Prepared by using Solid Reaction Method](#)
E. H. Sujiono, J. Agus, S. Samnur et al.



IOP | ebooks™

Bringing you innovative digital publishing with leading voices to create your essential collection of books in STEM research.

Start exploring the collection - download the first chapter of every title for free.

Influence of Annealing Time Variation on Crystal Structure and Morphology of Oxide Material $\text{Nd}_{1.2}\text{FeO}_3$ by Solid-State Reaction Method

E. H. Sujiono^{1,*)}, A.C. M. Said¹, M. Y. Dahlan¹, R. A. Imran¹, and S. Samnur¹

¹Laboratory 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, $\text{Nd}_{1.2}\text{FeO}_3$ 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 $0.2\mu\text{m}$. These results indicate the variation of annealing time 1, 2, and 3 hours did not significantly affect the crystallinity and morphology of $\text{Nd}_{1.2}\text{FeO}_3$ oxide material.

Keywords. Annealing time, crystallinity, morphology, $\text{Nd}_{1.2}\text{FeO}_3$ 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, $\text{Nd}_{1.2}\text{FeO}_3$ 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 $\text{Nd}_{1.2}\text{FeO}_3$ 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 $\text{Nd}_{1.2}\text{FeO}_3$ 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 $\text{Nd}_{1.2}\text{FeO}_3$ 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 $\text{Nd}_{1.2}\text{FeO}_3$ were characterized by using x-ray diffraction (Rigaku Miniflex II $\text{CuK}\alpha$, $\lambda = 0.154$ nm) 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 $\text{Nd}_{1.2}\text{FeO}_3$ 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 $2\theta = 32.5^\circ$ 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].

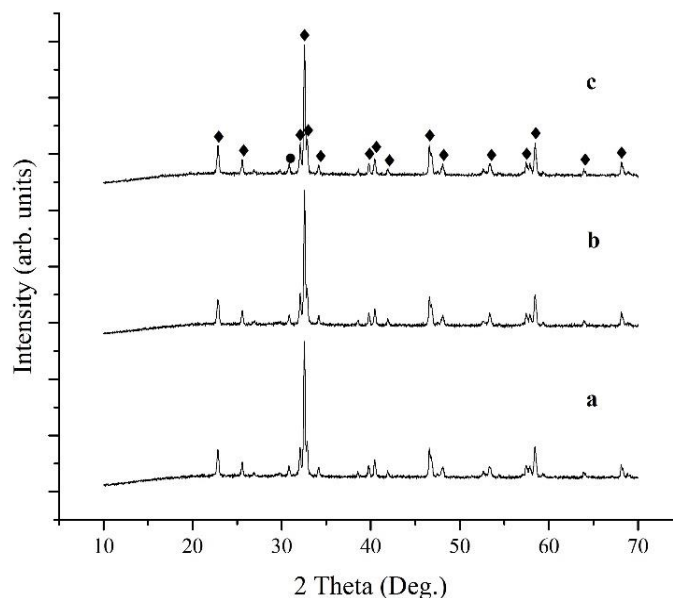


Figure 1. XRD pattern of $\text{Nd}_{1.2}\text{FeO}_3$ as variation of annealing time a = 1 h, b = 2 h, and c = 3 h, respectively (\blacklozenge = NdFeO_3 , \bullet = Nd_2O_3).

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 Xinhuet 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 $a = 5.58 \text{ \AA}$, $b = 7.76 \text{ \AA}$, $c = 5.45 \text{ \AA}$, 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 $950 \text{ }^\circ\text{C}$ 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).

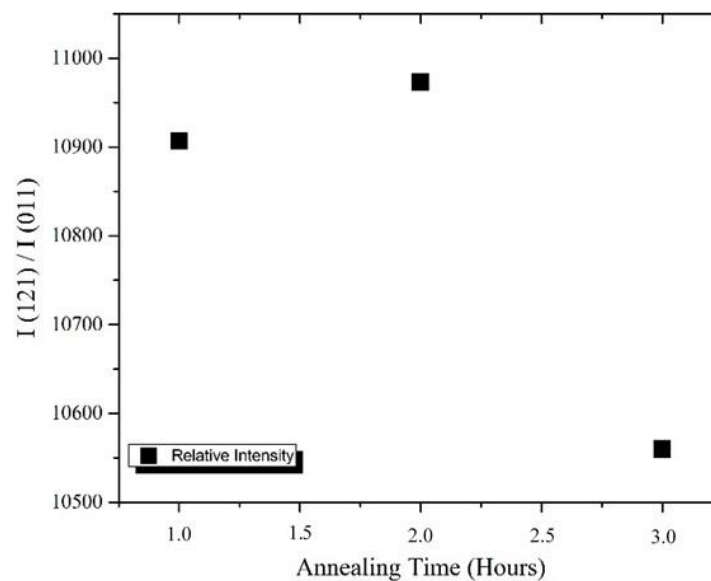


Figure 2. Curve comparison relative intensity of $Nd_{1.2}FeO_3$ (121) and Nd_2O_3 (011) for each variation of annealing time

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 $\text{Nd}_{1.2}\text{FeO}_3$ oxide material

Annealing time (h)	2θ (°)	FWHM (°)	Crystallite Size (nm)
1	32.56	0.22	1393.08 ± 0.021
2	32.58	0.22	1393.10 ± 0.021
3	32.58	0.22	1393.10 ± 0.021

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 $0.2 \mu\text{m}$. 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].

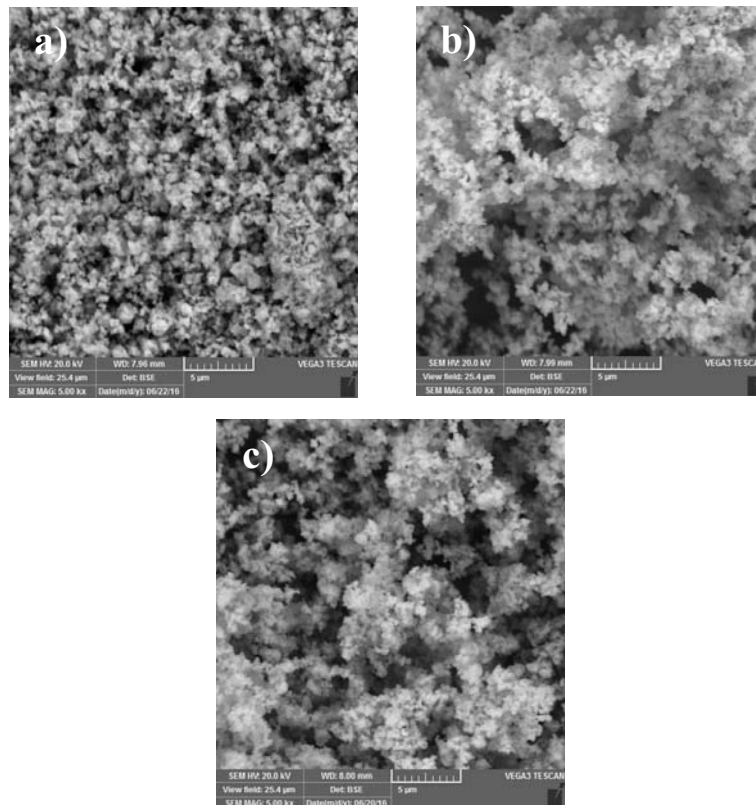


Figure 3. SEM micrograph of $\text{Nd}_{1.2}\text{FeO}_3$ as variation of annealing time t of (a) 1 h, (b) 2 h, and (c) 3 h, respectively.

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}FeO₃ 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}FeO₃ 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 YBa₂Cu₃O_{7- δ} 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 TiO₂ material that annealing variation affects the resistivity of the material [21].

4. Conclusions

Nd_{1.2}FeO₃ 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 32.5°. There were Nd_{1.2}FeO₃ and Nd₂O₃ 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 μ m. 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

This research was funded by Directorate research and Community Services, Ministry of Research, Technology and Higher Education, Republic of Indonesia, under research scheme of *Hibah Kompetensi* fiscal year 2017. The authors acknowledge this financial support.

References

- [1] Pena M A and Fierro J L G 2001 Chemical Structures and Performance of Perovskite Oxides *Chemical Reviews* **101** 1981 – 2018
- [2] Ho T G *et al* 2011 Nanosized perovskite oxide NdFeO₃ as material for a carbon-monoxide catalytic gas sensor *Advanced in Natural Science: Nanoscience and Nanotechnology* **2** 15012 – 15015
- [3] Taneja S *et al* 1993 Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms *Nuclear Instruments and Methods in Physics Research* **76** 127 – 129
- [4] Opuchovic O *et al* 2015 Sol-gel synthesis, characterization and application of selected sub-microsized lanthanide (Ce, Pr, Nd, Tb) ferrites *Dyes and Pigment*. **118** 176 – 182
- [5] Tongyun C *et al* 2012 NdFeO₃ as anode material for S/O₂ solid oxide fuel cells *Journal of Rare Earths*. **30** 1138 – 1141
- [6] Xinshu N *et al* 2003 *Journal of Rare Earths*. **21** 550 – 553
- [7] Ru Z *et al* 2010 Electrical and CO-sensing properties of NdFe_{1-x}Co_xO₃perovskite system *Journal of Rare Earths*. **28** 591 – 595
- [8] Motlagh M *et al* 2013 Chemical Synthesis and Characterization of Perovskite NdfeO3 Nanocrystals via a Co-Precipitation Method *International Journal of Nanoscience and Nanotechnology*. **9** 7 – 14

- [9] Song G *et al* 2015 Spin reorientation transition process in single crystal NdFeO₃ *Solid State Communication*. **211** 47 – 51
- [10] Chanda S, Saha S, and Sinha T 2013 Raman spectroscopy and dielectric properties of nanoceramic NdFeO₃ *Material Research Bulletin*. **48** 1688 – 1693
- [11] Pikula T *et al* 2014 Structure and hyperfine interactions in Bi_{1-x}Nd_xFeO₃ solid solutions prepared by solid-state sintering *Journal of Alloys and Compounds*. **606** 1 – 6
- [12] Belessi V C *et al* 1999 Structure and catalytic activity of La_{1-x}FeO₃ system ($x=0.00, 0.05, 0.10, 0.15, 0.20, 0.25, 0.35$) for the NO+CO reaction *Applied Catalysis A : General*. **177** 53 – 68
- [13] Eko H S 2008 *Paduan oksida logam Nd1(Fe)XBa2-XCu3OY dan metode pembuatannya* I. D. Patent No. P00200800471 (in Indonesia)
- [14] Eko H S *et al* 2001 Crystal Structure and Morphology Analysis of Nd_{1+x}Ba_{2-x}Cu₃O₇ Oxide Alloy Surface Developed by Solid State Reaction Method *Physica Status Solidi (A) Applied Research*. **187** 471 – 479
- [15] Bhagavannarayana G *et al* 2005 A study of the effect of annealing on Fe-doped LiNbO₃ by HRXRD, XRT and FT-IR *Journal of Applied Crystallography*. **38** 768 – 771
- [16] Zharvan V *et al* 2017 The Effect of Molar Ratio on Crystal Structure and Morphology of Nd_{1+x}FeO₃ ($X=0.1, 0.2, \text{ and } 0.3$) Oxide Alloy Material Synthesized by Solid State Reaction Method *IOP Conference: Materials Science and Engineering*. **202** 012072
- [17] Singh S *et al* 2013 Fabrication of nanobeads structured perovskite type neodymium iron oxide film: Its structural, optical, electrical and LPG sensing investigations *Sensors and Actuators B: Chemical*. **177** 730 – 739
- [18] Tighineanu A *et al* 2010 Conductivity of TiO₂ nanotubes: Influence of annealing time and temperature *Chemical Physics Letters*. **494** 260 – 263
- [19] Eko H S 2007 Optimization of Surface Smoothness of Nd 1.12 Ba 1.88 Cu₃O_{7-δ} Thin Films *Indonesian Journal of Physics*. **18** 107 – 110
- [20] Eko H S, Arifin P, and Barmawi M 2002 YBa₂Cu₃O_{7-δ} thin films deposited by a vertical MOCVD reactor *Materials Chemistry and Physics*. **73** 47 – 50
- [21] Shanker J, Suresh M B, and Babu D S 2015 Synthesis, Characterization and Electrical Properties of NdXO₃ ($X=Cr, Fe$) Nanoparticles *Materials Today: Proceedings*. **3** 2091 – 2100