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Refinement Analysis using the Rietveld Method of Nd_{1.2}Fe₁₀O₃ Oxide
Material Synthesized by Solid-State Reaction
E.H. Sujiono, A.CM. Said, M.Y. Dahlan, R.A. Imran, S. Samnur

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Dear Secretary of the editorial board
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Here presented correction of the Proof of article entitled:
Refinement Analysis using the Rietveld Method of Nd_{1.2}Fe₁O₃ Oxide Material Synthesized by Solid-State Reaction
E.H. Sujiono, A.C.M. Said, M.Y. Dahlan, R.A. Imran, S. Samnur

"Page 1, line 35. the word "S/O₂dan" change on "S/O₂ and".

"Page 2, line 25. the word "Nd_{1.2}FeO" change on "Nd_{1.2}Fe₁O₃"

"Page 2, line 51. the word "Table 1 – XRD analysis of Nd_{1.2}FeO₃ oxide material" move on to line 52 on top of Table 1.

"Page 2, line 52. the content of Table 1 should be change as an attachment file.

"Page 2, line 64. the word "Scherrer equation" change on "Scherrer equation (3.1):"

"Page 3, line 56. the word "NdFeO₃" change on "Nd_{1.2}Fe₁O₃"

"Page 3, line 68. the word "a = 5.580412 Å" change on "a = 5.580412 Å, "

"Page 4, line 2. the word "of each phase" change on "of Nd_{1.2}Fe₁O₃ phase"

"Page 4, line 58. the word "187 No. 2, 471 (2001)" change on "187, Issue 2 (2001) [https://doi.org/10.1002/1521-396X\(200110\)187:2<471: AID-PSSA471>3.0.CO;2-M](https://doi.org/10.1002/1521-396X(200110)187:2<471: AID-PSSA471>3.0.CO;2-M)"

"Page 4, line 59. the word "Math. Chem. Phys." change on "Materials Chemistry and Physics"

"Page 4, line 60. the word "73, 47 (2002)" change on "73, 1 (2002) [https://doi.org/10.1016/S0254-0584\(01\)00351-0](https://doi.org/10.1016/S0254-0584(01)00351-0)"

"Page 4, line 62. the word "Math. Sci. Eng. 202, 012072 (2017)" change on "Mater. Sci. Eng. 202, 012072 (2017) doi: 10.1088/1757-899X/202/1/012072 "

Refinement Analysis using the Rietveld Method of Nd_{1.2}FeO₃ Oxide Material Synthesized by Solid-State Reaction

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Neodymium Ferrite Oxide (Nd_{1.2}FeO₃) has been successfully synthesized using solid state reaction by varying annealing time. Structural crystallographic characteristics were obtained by X-ray diffraction. The results of X-ray diffraction analysis showed the samples had been identified composed of NdFeO₃ and Nd₂O₃ phase, with peak dominant correspond to *hkl* (121), FWHM value of 0.22° and estimated crystal size of 393 nm. Analysis using Rietveld methods obtained Nd_{1.2}FeO₃ oxide material has a crystal structure is orthorhombic with space-group of PNMA. The results is comparable as was reported elsewhere that the oxide material is useful for gas sensor application.

Keywords: Annealing time, The Lattice constant, Solid-state reaction, The Rietveld method.

DOI: [10.21272/jnep.10\(2\).02034](https://doi.org/10.21272/jnep.10(2).02034)

PACS numbers: 61.05.cp, 74.62.Bf, 81.05.Bx, 82.33.pt

1. INTRODUCTION

NdFeO₃ material has long attracted attention as a material that can be used as raw material gas sensor[1][2], fuel cells[3], the catalyst material gas sensor[4], and magnetic materials[5][6]. NdFeO₃ as a raw material gas sensor sensitive to some kind of gas. As research conducted by Niu Xinshu et al. (2003) showed that the nanocrystals NdFeO₃ could be used as H₂S gas sensor, which between selectivity and sensitivity of the sensor by H₂S concentrations have quite an interesting relation[1]. Additionally, the research carried out by Chen Tongyung et al. (2012) showed that NdFeO₃ could be to anode material S/O₂ dan SO₂/O₂-SOFCs[3]. While the research conducted by Truong Giang Ho et al. (2010) showed that the catalyst of the gas sensor NdFeO₃ material has good stability and sensitivity to CO gas [4].

Has been known a variety of ways to synthesize oxide materials, one of the most conventional ways that are easy to use is a solid state reaction method. This method was done by mixing different metal oxide alloys at high temperatures[7, 8].

Rietveld analysis was advanced analysis to find out the physical properties of a material quantitatively based on the XRD data. Rietveld analysis was a method that matches the theoretical curve with the experimental curve until both curves appropriate [9]. Both of curve was an order by using the least squares method was performed repeatedly (iteration), so there's a match between two curves then that data can be observed by the data calculation [10].

In this study, Nd_{1.2}FeO₃ materials have been synthesized using solid state reaction by varying the annealing time for 1, 2 and 3 hours at 450 °C, respectively. Satyendra Singh (2012), presented that the optimal temperature for annealing Nd_{1.2}FeO₃ material is at 450 °C because it would make the sample more responsive [11]. This annealing temperature is also has reported elsewhere for varied of oxide material which is YBaCuO and NdFeO[12 13, 14]. The sample obtained

and then characterized by X-ray diffraction to identify the phase has formed. The lattice constant value will be performed with refinement analysis using Rietveld methods based on the results of X-ray diffraction characterization.

2. EXPERIMENTAL

NdFeO₃ material synthesized by using solid-state reaction method. Synthesis process begins by mixing of raw material Nd₂O₃ 99.99% (*Strem Chemicals*) and Fe₂O₃ 99.99% (*Sigma-Aldrich*) in accordance with stoichiometry calculations. In the first stage, the mixture of Nd₂O₃ and Fe₂O₃ grinded for ± 3 hours, then calcined at 700 °C for 6 hours. The material then grinded back for ± 5 hours and then sintered at 950 °C for 6 hours.

In the second stage, the sample was re-grinded for ± 3 hours, then calcined at 950 °C for 6 hours. Then grinded the sample back for ± 5 hours, then sintering at 950 °C, by varying the annealing time for 1, 2 and 3 hours respectively.

The obtained Nd_{1.2}FeO₃ oxide material has been synthesized and then characterized using x-ray diffraction (Rigaku Mini Flex II CuKα, λ = 0.154 nm) to find out phase formed. The results of x-ray diffraction characterization were then analyzed using the Rietveld method.

Refinement analysis begins by creating a sample database with regard phase and the atoms that make up phase, as well as the radiation source, used to characterize the sample. In addition, the space group determine in accordance with the sample into one of the parameters that must be observed. In this research space group in accordance with the sample was PNMA. Calculation of refinement analysis using Rietveld method began by matching the background between the theoretical curve and the experimental curve. Then continued with match the peak of the curve by adjusting the scale phase and the lattice parameter of the sample in Phases menu. Then on the menu Histogram,

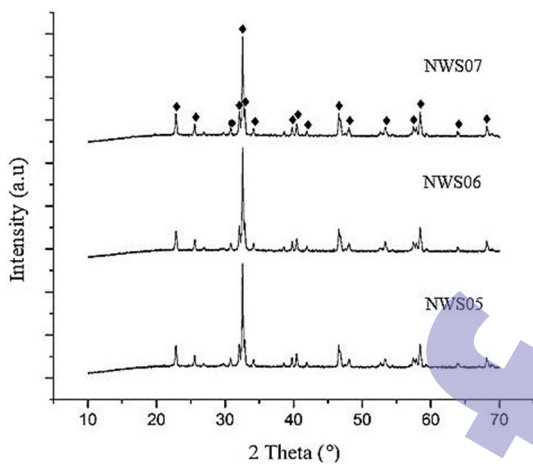
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1 which must be considered is the value of the parameter
 2 zero which is a 2θ correction instrument. Sample dis-
 3 placement which is the amount of inaccuracy in the
 4 measurement of the vertical position of the sample as
 5 well as the parameters of B^{-1} , B^0 , B^1 , and B^2 affecting
 6 the high peaks of the sample. Then the shape of the
 7 curve will be refined on the Sample menu. In this
 8 section, the shape and width of the peak will be refined
 9 through a U-Gaussian parameter, the Lorentzian para-
 10 meter (size) and asymmetry. While the peak tail in-
 11 fluenced by Lorentzian parameters (size). Refinement
 12 results can be seen at Information tab in the menu
 13 Output.

15 3. RESULTS AND DISCUSSION

17 3.1 Analysis of X-Ray Diffraction

19 The results of x-ray diffraction characterization of
 20 $Nd_{1.2}FeO_3$ the sintering at a temperature of $950\text{ }^\circ\text{C}$ is
 21 shown in Figure 1.



23 Fig. 1 – XRD pattern of $Nd_{1.2}FeO_3$ powder in the variation of
 24 annealing time 1 hour, 2 hours and 3 hours, respectively (● =
 25 Nd_2O_3 ◆ = $NdFeO_3$)

27 In Figure 1 shows that in this study, there are two
 28 phases, the dominant phase $NdFeO_3$ (diamond) and
 29 phase Nd_2O_3 (circle). The $NdFeO_3$ material has formed
 30 a crystalline phase with the highest peak being on the
 31 plane (121). This was according to Yabin Wang et al.
 32 research (2010), which is known that the peak (121)
 33 was the most sensitive of peak to certain gases [12].
 34 Results found equally to the research conducted by
 35 NiuXinshu et al. (2003) who found the peak (121) at
 36 $2\theta = 32.5^\circ$ [1]. This pattern also indicated that the
 37 $NdFeO_3$ material has a crystalline structure ortho-
 38 rhombic perovskite type. As has reported by previous
 39 researchers that the material crystal structure ortho-
 40 rhombic perovskite-type was a material that can be
 41 used as raw material gas sensor [1, 4, 11].

42 The results of X-ray diffraction analysis of the ma-
 43 terial $Nd_{1.2}Fe_1O_3$ can be seen from Table 1.

44 Table 1 shown the $NdFeO_3$ phase has formed at a
 45 2θ angle of 32.5° with the peak intensity reached 13200
 46 counts. In accordance with research Niu Xinshu, peaks
 47 at an angle 2θ of 32.5° were identified as hkl (121),
 48 which is the most sensitive peak to some specific
 49 Table 1 – XRD analysis results of $Nd_{1.2}FeO_3$ oxide material

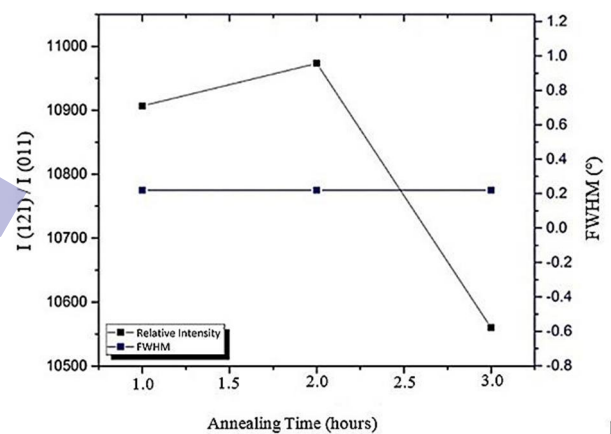
| Annealing time | 2θ ($^\circ$) | Peak Int. | FWHM | Crystal size (nm) |
|----------------|------------------------|-----------|------|-------------------|
| 1 jam | 32.56 | 13286.6 | 0.22 | 393.08 ± 0.02 |
| 2 jam | 32.58 | 7 | 0.22 | |
| 3 jam | 32.58 | 13233.3 | 0.22 | 393.10 ± 0.02 |
| | | 3 | | |
| | | 12873.3 | | 393.10 ± 0.02 |
| | | 3 | | |

53 types of gas [1]. FWHM value is an indication of the
 54 crystalline quality of the oxide material. The smaller
 55 FWHM value of the crystal means that the material
 56 quality more better [12-14]. Furthermore, FWHM val-
 57 ues also indicate the level of homogeneity of materials.
 58 We found that in this study the FWHM value is similar;
 59 indicate that the variation annealing time does not
 60 affect the level crystal quality and the homogeneity of
 61 $Nd_{1.2}FeO_3$ oxide material.

62 The crystal size can be estimated using Debye
 63 Scherrer equation:

$$D = \frac{0.89\lambda}{B} \cos \theta \tag{3.1}$$

64 Where λ was the wavelength of the X-ray
 65 (1.54056 \AA), θ was the Bragg angle and B was FWHM.
 66 By applying of Debye Scherrer formula obtained crystal
 67 size for all samples is 393 nm, an indication that the
 68 oxide material has been studied is in categories of mi-
 69 cro-material [16].



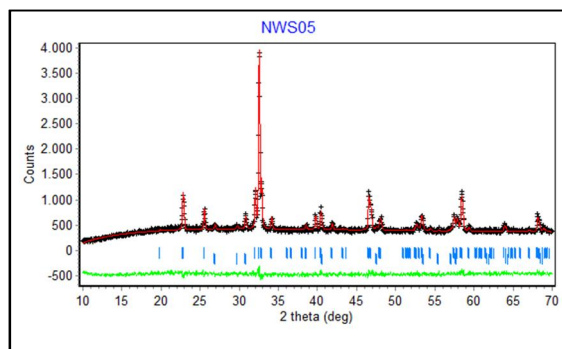
74 Fig. 2 – Comparison curve between the relative intensity and
 75 FWHM values for each variation of annealing time

76 Figure 2 shows the relative intensity, FWHM as a
 77 function of variation of annealing time. Relative
 78 intensity, in this case, was the ratio between the high-
 79 est intensity for both the phase obtained, which is
 80 phase $NdFeO_3$ and phase Nd_2O_3 . The highest peak
 81 phase $NdFeO_3$ is corresponding to hkl (121). Mean-
 82 while, the highest peak on the phase of Nd_2O_3 is re-
 83 lated to (011). In this study appearing of the Nd_2O_3
 84 peak due to lack of grinding process before calcina-
 85 tion treatment. It is can be caused the Nd_2O_3 less
 86 reacted with Fe_2O_3 phase to obtain formation of
 87 $NdFeO_3$ phase [2]. In contrast, decreasing peak of
 88 (011) will be correlated with increasing the intensity
 89 of the peak (121) and this data as an indication that
 90 the crystal quality of oxide material is improving. These
 91 results

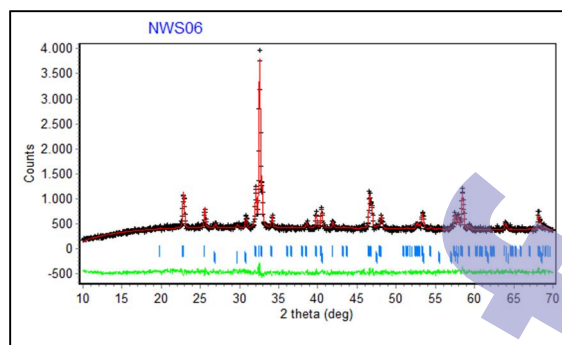
1 indicate that the variation of annealing time has a 33
2 significant change of relative intensity of peaks (121) 34
3 and (011), while the value of FWHM and crystal size 35
4 has been obtained is similar. 36

6 3.2 Rietveld Analysis

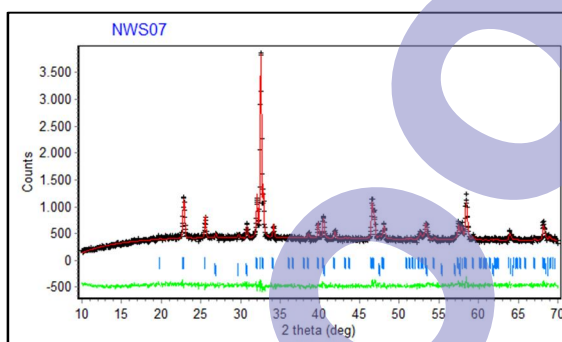
7 Advanced analysis of data from X-ray diffraction 38
8 characterization was to use Rietveld method. In this 39
9 study used Rietica software for smoothing data from 40
10 X-ray diffraction characterization. 41
11
12



(a)



(b)



(c)

13
14
15
16
17
18
19
20
21
22 Fig. 3 – Refinement results of NdFeO_3 oxide material using 68
23 the Rietveld method: (a) NWS05; (b) NWS06; and (c) NWS07, 69
24 respectively. 70

25 Refinement analysis results showed that the samples 72
26 of $\text{Nd}_{1.2}\text{FeO}_3$ have an orthorhombic crystal structure 73
27 with space group PNMA. This is according to re- 74
28 search conducted by Sadhan Chanda et al. [17]. 75

29 The observed data are indicated by pluses (+) and 76
30 the calculated data by the solid line overlying them. 77

31 The lower curve shows the difference between the 78
32 79

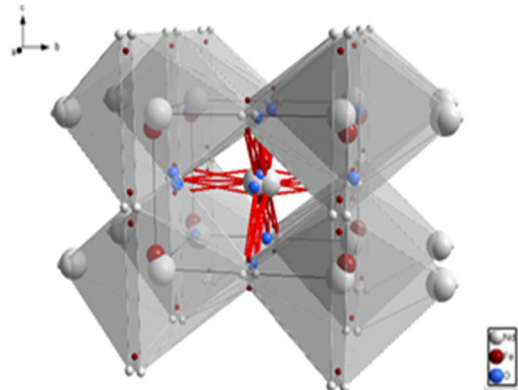
observed and calculated diffraction patterns (red). The
success of refinement a sample was not only seen of a
match between the theoretical curve and the experi-
mental curve, that could only be observed visually, as
shown in figure 3. But also seen from the GoF resulting
from the refinement. If the value of GoF was below 4
then refinement considered successful [18]. The Data of
Rietveld refinement results can be seen in Table 2.

42 Table 2 – The Data of Rietveld refinement results using soft-
43 ware Rietica based on XRD analysis 44

| Annealing Time (hours) | Rp (%) | Rwp (%) | Rexp (%) | GoF (%) |
|--|--------|---------|----------|---------|
| 1 | 4.50 | 6.06 | 6.43 | 0.8899 |
| 2 | 4.73 | 6.15 | 6.38 | 0.9303 |
| 3 | 4.67 | 6.06 | 6.37 | 0.9053 |
| $^{\text{a}}\text{NdFeO}_3$ (Jada Shanker) | 8.6 | 6.1 | 10.9 | 1.6 |

45 The data in Table 2 shows the GoF parameter ob-
46 tained from the refinement results has been studied in
47 this research and with the comparison, refinement is
48 performed by Jada Shanker et al. [19]. 49

50 Several results of the analysis can be read directly
51 from the output data Rietveld analysis is the lattice pa-
52 rameter, the percentage of samples molar and sample
53 displacement. The lattice parameters obtained that can be
54 used to describe the location of atoms in the crystal
55 structure of materials. Table 3 shows the molar percentage
56 of the NdFeO_3 phase of each sample, ranging from 99 %.
57 The value tend continues to increase with the heating
58 time given. Molar percentage obtained show more accurate
59 results than the traditional way. While the sample dis-
60 placement indicates the value inaccuracy in the meas-
61 urement sample vertical position, meaning the value close
62 to 0 is the inaccuracies tend to be smaller. 63



64
65
66 Fig. 4 – The visualization results of the crystal structure of
67 $\text{Nd}_{1.2}\text{FeO}_3$ based on data from refinement using Rietveld
68 method (with $a = 5.580412 \text{ \AA}$ $b = 7.758973 \text{ \AA}$, $c = 5.449359 \text{ \AA}$) 69

70 Lattice parameters obtained from the refinement
71 can be used to describe the location of atoms in the
72 crystal structure NdFeO_3 which can be seen in Fig-
73 ure 4. Figure 4 shows the orthorhombic crystal struc-
74 ture of NdFeO_3 oxide material, where Nd atoms
75 indicate gray color, Fe atom indicates red color, and O
76 atom indicates blue color. While the bonds between the
77 atoms are identified as a covalent bond. 78
79

Table 3 – The refinement result using software Rietica to analyze molar percentage of each phase, the lattice parameter, and sample displacement

| Annealing Time | Molar Percentage | Lattice Parameter | | | Sample Displacement |
|----------------|------------------|-------------------|-------------------|-------------------|---------------------|
| | | A (Å) | B (Å) | C (Å) | |
| 1 h | 99.87 % | 5.581260±0.000682 | 7.759268±0.000888 | 5.448154±0.000616 | - 0.067404 |
| 2 h | 99.93 % | 5.580412±0.000704 | 7.758973±0.000919 | 5.449359±0.000634 | - 0.100198 |
| 3 h | 99.93 % | 5.580855±0.000712 | 7.760073±0.000924 | 5.448353±0.000644 | - 0.100198 |

4. CONCLUSION

NdFeO₃ synthesis using solid state reaction method has been successfully studied. The X-ray diffraction analysis obtained of two dominant phase which is Nd_{1.2}Fe₁O₃ oxide material and Nd₂O₃. The results also found that the highest peak is corresponding to *hkl* (121) which is known that peak as a sensitive to various gases. The FWHM value is 0.22° with an estimated crystals size of 393 . The refinement analysis using Rietveld method obtained the crystal structure of the

material NdFeO₃ was orthorhombic with space group PNMA. Therefore, NdFeO₃ oxide material obtained in this study can be used as raw material for a gas sensor.

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Thank you for your kind cooperation.

With best regards,

Eko Hadi Sujiono

Corresponding author.

[Kutipan teks disembunyikan]



Table 1-XRD analysis results of Nd_{1.2}FeO₃ oxide material.docx
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Table 1-XRD analysis results of Nd_{1.2}FeO₃ oxide material.docx
13K

Table 1 – XRD analysis results of Nd_{1.2}FeO₃ oxide material

| Annealing time (hours) | 2θ (°) | Peak Int. (cps) | FWHM (°) | Crystal size (nm) |
|-------------------------------|---------------|------------------------|-----------------|--------------------------|
| 1 | 32.56 | 13286.67 | 0.22 | 393.08±0.02 |
| 2 | 32.58 | 13233.33 | 0.22 | 393.10±0.02 |
| 3 | 32.58 | 12873.33 | 0.22 | 393.10±0.02 |

Refinement Analysis using the Rietveld Method of Nd_{1.2}Fe₁O₃ Oxide Material Synthesized by Solid-State Reaction

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Neodymium Ferrite Oxide (Nd_{1.2}FeO₃) has been successfully synthesized using solid state reaction by varying annealing time. Structural crystallographic characteristics were obtained by x-ray diffraction. The results of x-ray diffraction analysis showed the samples had been identified composed of NdFeO₃ and Nd₂O₃ phase, with peak dominant correspond to *hkl* (121), FWHM value of 0.22° and estimated crystal size of 393 nm. Analysis using Rietveld methods obtained Nd_{1.2}FeO₃ oxide material has a crystal structure is orthorhombic with space-group of PNMA. The lattice constant value for each variation parameter is NWS05 a= |5.581260±0.000682|Å, b=|7.759268±0.000888|Å, c=|5.448154±0.000616|Å, for NWS06 a=|5.580412±0.000704|Å, b=|7.758973±0.000919|Å, c=|5.449359±0.000634|Å, and for NWS07 a=|5.580855±0.000712|Å, b=|7.760073±0.000924|Å, c=|5.448353±0.000644|Å, respectively with estimated value *Goodness of Fit* (GoF) has obtained is 0.9%. This result is comparable as was reported elsewhere that the oxide material is useful for gas sensor application.

Keywords: annealing time, the lattice constant, solid-state reaction, the Rietveld method.

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81.05.Bx, 82. 33. pt

1. INTRODUCTION

NdFeO₃ material has long attracted attention as a material that can be used as raw material gas sensor[1][2], fuel cells[3], the catalyst material gas sensor[4], and magnetic materials[5][6]. NdFeO₃ as a raw material gas sensor sensitive to some kind of gas. As research conducted by Niu Xinshu et al. (2003) showed that the nanocrystals NdFeO₃ could be used as H₂S gas sensor, which between selectivity and sensitivity of the sensor by H₂S concentrations have quite an interesting relation[1]. Additionally, the research carried out by Chen Tongyung et al. (2012) showed that NdFeO₃ could be to anode material S/O₂-dan SO₂/O₂-SOFCs[3]. While the research conducted by Truong Giang Ho et al. (2010) showed that the catalyst of the gas sensor NdFeO₃ material has good stability and sensitivity to CO gas[4].

Has been known a variety of ways to synthesize oxide materials, one of the most conventional ways that are easy to use is a solid state reaction method. This method was done by mixing different metal oxide alloys at high temperatures[7][8].

Rietveld analysis was advanced analysis to find out the physical properties of a material quantitatively based on the XRD data. Rietveld analysis was a method that matches the theoretical curve with the experimental curve until both curves appropriate[9]. Both of curve was an order by using the least squares method was performed repeatedly (iteration), so there's a match between two curves then that data can be observed by the data calculation[10].

In this study, Nd_{1.2}FeO₃ materials have been synthesized using solid state reaction by varying the annealing time for 1, 2 and 3 hours at 450°C, respectively. Satyendra Singh (2012), presented that the optimal temperature for annealing Nd_{1.2}FeO₃-material is at 450°C because it would make the sample more responsive[11]. This annealing temperature is

also has reported elsewhere for varied of oxide material which is YBaCuO and NdFeO₃[12, 13, 14]. The sample obtained and then characterized by x-ray diffraction to identify the phase has formed. The lattice constant value will be performed with refinement analysis using Rietveld methods based on the results of x-ray diffraction characterization.

2. EXPERIMENTAL

NdFeO₃ material synthesized by using solid-state reaction method. Synthesis process begins by mixing of raw material Nd₂O₃ 99.99% (*Strem Chemicals*) and Fe₂O₃ 99.99% (*Sigma-Aldrich*) in accordance with stoichiometry calculations. In the first stage, the mixture of Nd₂O₃ and Fe₂O₃ grinded for ± 3 hours, then calcined at 700°C for 6 hours. The material then grinded back for ± 5 hours and then sintered at 950°C for 6 hours.

In the second stage, the sample was re-grinded for ± 3 hours, then calcined at 950°C for 6 hours. Then grinded the sample back for ± 5 hours, then sintering at 950°C, by varying the annealing time for 1, 2 and 3 hours respectively.

The obtained Nd_{1.2}FeO₃ oxide material has been synthesized and then characterized using x-ray diffraction (Rigaku Mini Flex II CuKα, λ = 0.154 nm) to find out phase formed. The results of x-ray diffraction characterization were then analyzed using the Rietveld method.

Refinement analysis begins by creating a sample database with regard phase and the atoms that make up phase, as well as the radiation source, used to characterize the sample. In addition, the space group determine in accordance with the sample into one of the parameters that must be observed. In this research space group in accordance with the sample was PNMA. Calculation of refinement analysis using Rietveld method began by matching the background between the

theoretical curve and the experimental curve. Then continued with match the peak of the curve by adjusting the scale phase and the lattice parameter of the sample in *Phases* menu. Then on the menu *Histogram*, which must be considered is the value of the parameter *zero* which is a 2θ correction instrument. *Sample displacement* which is the amount of inaccuracy in the measurement of the vertical position of the sample as well as the parameters of *B-1*, *B0*, *B1*, and *B2* affecting the high peaks of the sample. Then the shape of the curve will be refined on the *Sample* menu. In this section, the shape and width of the peak will be refined through a *U-Gaussian parameter*, the *Lorentzian parameter (size)* and *asymmetry*. While the peak tail influenced by Lorentzian parameters (size). Refinement results can be seen at *Information* tab in the menu *Output*.

3. RESULTS AND DISCUSSION

3.1 Analysis of X-Ray Diffraction

The results of x-ray diffraction characterization of $Nd_{1.2}FeO_3$ the sintering at a temperature of $950^\circ C$ is shown in Figure 1.

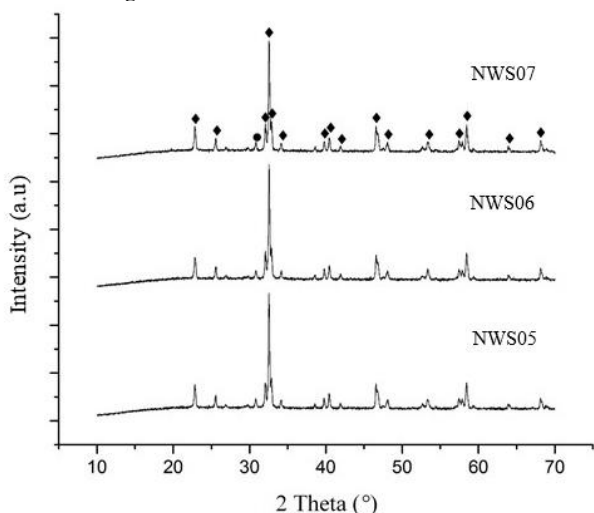


Fig. 1 – XRD pattern of $Nd_{1.2}FeO_3$ powder in the variation of annealing time 1 hour, 2 hours and 3 hours, respectively (● = Nd_2O_3 ♦ = $NdFeO_3$)

In Figure 1 shows that in this study, there are two phases, the dominant phase $NdFeO_3$ (diamond) and phase Nd_2O_3 (circle). The $NdFeO_3$ material has formed a crystalline phase with the highest peak being on the plane (121). This was according to Yabin Wang et al. research (2010), which is known that the peak (121) was the most sensitive of peak to certain gases[12]. Results found equally to the research conducted by NiuXinshu et al. (2003) who found the peak (121) at $2\theta = 32.5^\circ$ [1]. This pattern also indicated that the $NdFeO_3$ material has a crystalline structure orthorhombic perovskite type. As has reported by previous researchers that the material crystal structure orthorhombic perovskite-type was a material that can be used as raw material gas sensor[1][4][11].

The results of x-ray diffraction analysis of the material $Nd_{1.2}Fe_1O_3$ can be seen from Table 1.

Table 1 – XRD analysis results of $Nd_{1.2}FeO_3$ oxide material

| Annealing time (hours) | 2θ ($^\circ$) | Peak Int. (Counts) | FWHM ($^\circ$) | Crystal size (nm) |
|------------------------|------------------------|--------------------|-------------------|-------------------|
| 1 | 32.56 | 13286.67 | 0.22 | 393.08±0.02 |
| 2 | 32.58 | 13233.33 | 0.22 | 393.10±0.02 |
| 3 | 32.58 | 12873.33 | 0.22 | 393.10±0.02 |

Table 1 shown the $NdFeO_3$ phase has formed at a 2θ angle of 32.5° with the peak intensity reached 13200 counts. In accordance with research Niu Xinshu, peaks at an angle 2θ of 32.5° were identified as *hkl* (121), which is the most sensitive peak to some specific types of gas[1]. FWHM value is an indication of the crystalline quality of the oxide material. The smaller FWHM value of the crystal means that the material quality more better[12, 13, and 14]. Furthermore, FWHM values also indicate the level of homogeneity of materials. We found that in this study the FWHM value is similar; indicate that the variation annealing time does not affect the level crystal quality and the homogeneity of $Nd_{1.2}FeO_3$ oxide material.

The crystal size can be estimated using Debye Scherrer equation [16]:

$$D = \frac{0.89\lambda}{B} \cos\theta \quad (3.1)$$

Where λ was the wavelength of the x-ray (1.54056\AA), θ was the Bragg angle and B was FWHM. By applying of Debye Scherrer formula obtained crystal size for all samples is 393 nm, an indication that the oxide material has been studied is in categories of micro-material[17].

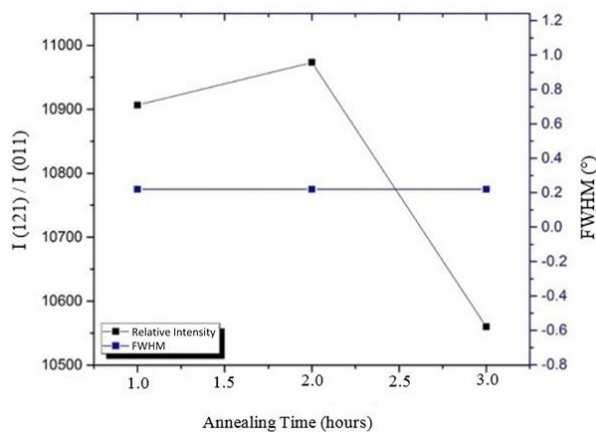


Fig. 2 – Comparison curve between the relative intensity and FWHM values for each variation of annealing time.

Figure 2 shows the relative intensity, FWHM as a function of variation of annealing time. Relative intensity, in this case, was the ratio between the highest intensity for both the phase obtained, which is phase $NdFeO_3$ and phase Nd_2O_3 . The highest peak phase $NdFeO_3$ is corresponding to *hkl* (121). Meanwhile, the highest peak on the phase of Nd_2O_3 is related to (011). In this study appearing of the Nd_2O_3 peak due to lack of grinding process before calcination treatment. It is can be caused the Nd_2O_3 less reacted with Fe_2O_3 phase to obtain formation of $NdFeO_3$ phase

[2]. In contrast, decreasing peak of (011) will be correlated with increasing the intensity of the peak (121) and this data as an indication that the crystal quality of oxide material is improving. These results indicate that the variation of annealing time has a significant change of relative intensity of peaks (121) and (011), while the value of FWHM and crystal size has been obtained is similar.

3.2 Rietveld Analysis

Advanced analysis of data from x-ray diffraction characterization was to use Rietveld method. In this study used Rietica software for smoothing data from x-ray diffraction characterization.

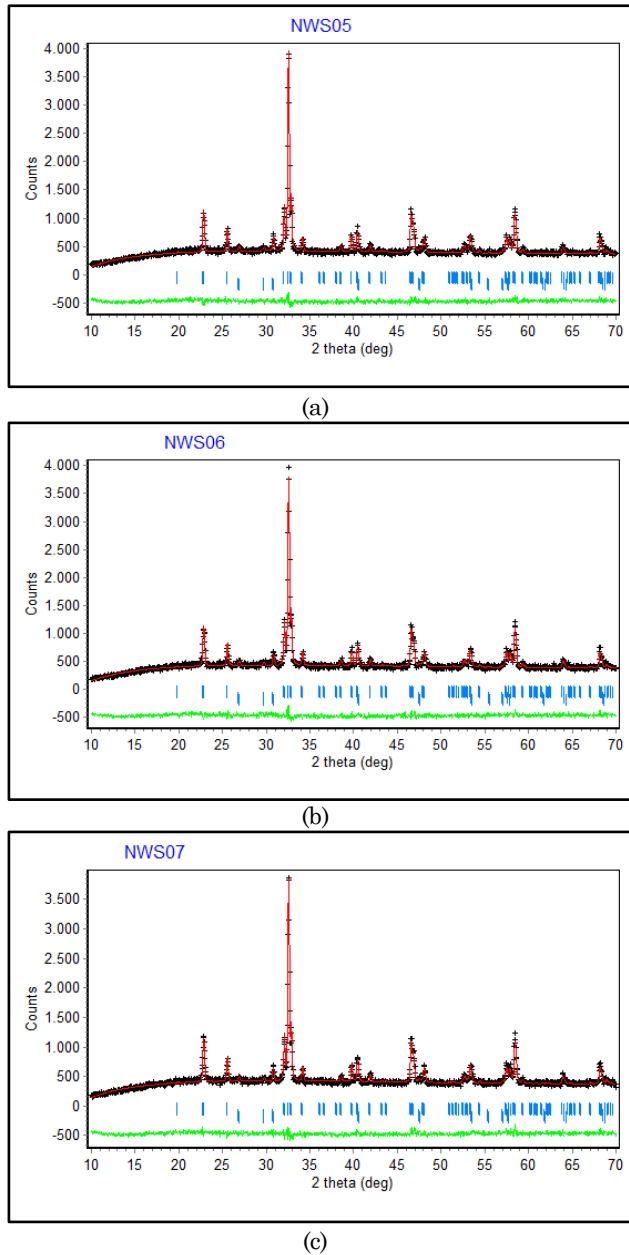


Fig. 3 – Refinement results of NdFeO₃ oxide material using the Rietveld method: (a) NWS05; (b) NWS06; and (c) NWS07, respectively.

Refinement analysis results showed that the sam-

ples of Nd_{1.2}FeO₃ have an orthorhombic crystal structure with space group PNMA. This is according to research conducted by Sadhan Chanda et al. (2013)[18].

The observed data are indicated by pluses (+) and the calculated data by the solid line overlying them. The lower curve shows the difference between the observed and calculated diffraction patterns (red). The success of refinement a sample was not only seen of a match between the theoretical curve and the experimental curve, that could only be observed visually, as shown in figure 3. But also seen from the GoF resulting from the refinement. If the value of GoF was below 4 then refinement considered successful[19]. The Data of Rietveld refinement results can be seen in Table 2.

Table 2 – The Data of Rietveld refinement results using software Rietica based on XRD analysis.

| Annealing Time (hours) | Rp (%) | Rwp (%) | Rexp (%) | GoF (%) |
|--|--------|---------|----------|---------|
| 1 | 4.50 | 6.06 | 6.43 | 0.8899 |
| 2 | 4.73 | 6.15 | 6.38 | 0.9303 |
| 3 | 4.67 | 6.06 | 6.37 | 0.9053 |
| ^a NdFeO ₃ (Jada Shanker) | 8.6 | 6.1 | 10.9 | 1.6 |

^areference

The data in Table 2 shows the GoF parameter obtained from the refinement results has been studied in this research and with the comparison, refinement is performed by Jada Shanker et al. (2016)[20].

Several results of the analysis can be read directly from the output data Rietveld analysis is the lattice parameter, the percentage of samples molar and sample displacement. The lattice parameters obtained that can be used to describe the location of atoms in the crystal structure of materials. Table 3 shows the molar percentage of the NdFeO₃ phase of each sample, ranging from 99%. The value tend continues to increase with the heating time given. Molar percentage obtained show more accurate results than the traditional way. While the sample displacement indicates the value inaccuracy in the measurement sample vertical position, meaning the value close to 0 is the inaccuracies tend to be smaller.

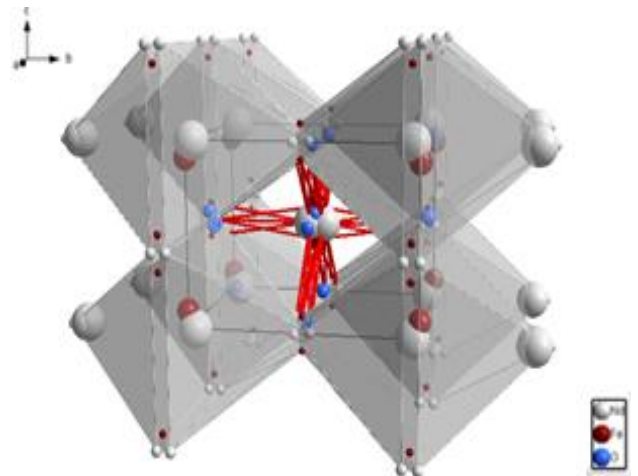


Fig. 4 – The visualization results of the crystal structure of Nd_{1.2}Fe₁O₃ based on data from refinement using Rietveld method (with a = 5.580412Å b = 7.758973Å, c = 5.449359Å).

Table 3 – The refinement result using software Rietica to analyze molar percentage of each phase, the lattice parameter, and sample displacement

| Annealing Time | Molar Percentage | Lattice Parameter | | | Sample Displacement |
|----------------|------------------|-------------------|-------------------|-------------------|---------------------|
| | | A (Å) | B (Å) | C (Å) | |
| 1 h | 99.87% | 5.581260±0.000682 | 7.759268±0.000888 | 5.448154±0.000616 | -0.067404 |
| 2 h | 99.93% | 5.580412±0.000704 | 7.758973±0.000919 | 5.449359±0.000634 | -0.100198 |
| 3 h | 99.93% | 5.580855±0.000712 | 7.760073±0.000924 | 5.448353±0.000644 | -0.100198 |

Lattice parameters obtained from the refinement can be used to describe the location of atoms in the crystal structure NdFeO₃ which can be seen in Figure 4. Figure 4 shows the orthorhombic crystal structure of NdFeO₃ oxide material, where Nd atoms indicate gray color, Fe atom indicates red color, and O atom indicates blue color. While the bonds between the atoms are identified as a covalent bond.

4. CONCLUSION

NdFeO₃ synthesis using solid state reaction method has been successfully studied. The X-ray diffraction analysis obtained of two dominant phase which is Nd_{1.2}Fe₁O₃ oxide material and Nd₂O₃. The re-

sults also found that the highest peak is corresponding to *hkl* (121) which is known that peak as a sensitive to various gases. The FWHM value is 0.22° with an estimated crystals size of 393 nm. The refinement analysis using Rietveld method obtained the crystal structure of the material NdFeO₃ was orthorhombic with space group PNMA. Therefore, NdFeO₃ oxide material obtained in this study can be used as raw material for a gas sensor.

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LAPORAN TAHUN TERAKHIR
PENELITIAN BERBASIS KOMPETENSI



PENGEMBANGAN SENYAWA OKSIDA NdFeO_3 DAN APLIKASINYA
DALAM PEMBUATAN SENSOR GAS

Tahun ke 2 dari rencana 2 tahun

Ketua dan Anggota Tim

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UNIVERSITAS NEGERI MAKASSAR

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Nomor: 1771/UN36.9/PL/2017

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DAN APLIKASINYA
DALAM PEMBUATAN SENSOR GAS

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RINGKASAN

Program penelitian ini direncanakan dalam masa 2 tahun untuk mencapai target penentuan parameter proses yang paling baik dalam pembuatan bahan paduan oksida NdFeO₃ untuk pengkajian sifat fisis dan mekanis mengenai bahan ini. Pembuatan sampel berbentuk bulk (pellet) dilakukan dengan menggunakan metode reaksi padatan (solid state reaction) di laboratorium Fisika Material Jurusan Fisika UNM. Penentuan parameter proses dapat dilakukan dengan mengoptimasi temperatur kalsinasi dan sintering, serta fraksi rasio molar dalam paduan NdFeO₃ dan modus pencampuran kering untuk induksi paladium.

Metode yang digunakan dalam penelitian ini adalah metode reaksi padatan (solid state reaction) dimana campuran bahan padatan dalam bentuk serbuk (powder) ditempatkan pada mortar dari bahan keramik dan dilakukan penggerusan sedemikian sehingga diperoleh campuran bahan yang homogen, sedangkan stoikiometri dari paduan ditentukan dengan menggunakan metode perhitungan molar dari unsur kimia bahan. Dalam bahan paduan NdFeO₃, untuk memperoleh bahan dengan variasi yang berbeda diberikan variasi temperatur (kalsinasi, sintering dan annealing) pada proses sintesis. Selanjutnya untuk induksi paladium pada senyawa NdFeO₃ dilakukan modus pencampuran kering.

Selanjutnya pada sampel yang lain dilakukan variasi untuk suhu dan waktu sintering. Perubahan sifat bahan dapat dianalisis berdasarkan hasil karakterisasi struktur dengan XRD (X-Ray Diffraction) dan morfologi dengan SEM (Scanning Electron Microscope), serta komposisi molar dengan EDAX (Energy Dispersive X-ray) (E. H. Sujiono dkk, 2001, 2002, 2005, 2009, dan 2011). Dari pengukuran tersebut dapat diperoleh data orientasi pertumbuhan kristal, struktur permukaan (morfologi) dan komposisi bahan, yang diukur untuk setiap variasi proses sintesis yang dilakukan. Pengukuran SEM, EDAX dan XRD dilakukan di laboratorium uji bahan Universitas Negeri Makassar (UNM).

Pada tahun pertama 2016, telah berhasil ditemukannya parameter proses terbaik untuk penumbuhan paduan NdFeO₃, bubuk paduan NdFeO₃ dan hasilnya antara lain: (1) dua artikel yang telah dipresentasikan pada 2 (dua) forum konferensi internasional yakni International Conference on Applied Material Science and Technology (ICAMST 2016) dan International Conference on Mathematics and Natural Science (ICMNS 2016); (2) dua artikel dikirim untuk dipublikasikan pada prosiding internasional terindeks Scopus, yaitu: satu diterbitkan pada Material Science and Engineering (IOP Proceeding) terindeks scopus dan satu artikel telah diterima dan siap untuk dipublikasikan pada Journal of Physics (IOP) terindeks scopus; (3) *invited speaker* pada konferensi internasional *One Asia Lecture Series* di Phnom Penh, *invited speaker* pada *the Second International Conference on Mathematics, Science, Technology, Education, and their Application* (2nd ICMSTEA 2016), dan *invited speaker* pada kuliah umum *One Asia Foundation* di Universitas Tadulako Palu; (4) satu artikel telah dipublikasikan pada jurnal terakreditasi nasional JPFI 12 (2) (2016) 177-182 dengan nomor DOI: 10.15294/jpfi.v12i2.4728. Selanjutnya parameter proses yang ditemukan telah digunakan untuk penelitian lanjutan tahun 2017.

Pada tahun kedua 2017, telah dilakukan penelitian sintesis senyawa oksida NdFeO₃ dengan doping Pd (Palladium) sebesar maksimum 30 wt%. Proses ini dilakukan untuk menambah sensitivitas NdFeO₃ sebagai bahan dasar pembuatan sensor gas. Luaran yang dicapai pada penelitian tahun 2017 adalah (1) Artikel yang diterbitkan pada Jurnal internasional/

internasional bereputasi antara lain: (a) *submitted article (status in review)* pada Crystal Research & Technology penerbit Wiley VCH Verlag GmbBH & Co, Germany terindeks SJR Q2, Scopus, dan Thomson Reuter Web of Science; (b) *submitted article* pada Journal of Nano- and Electronic Physics, Publisher Sumy Sate University terindeks SJR Q3 dan Scopus; (c) artikel yang diterbitkan pada Material Science and Engineering (IOP Proceeding) terindeks scopus DOI: 10.1088/1757-899X/202/1/012072 dan Journal of Physics (IOP) terindeks scopus DOI: 10.1088/1742-6596/846/1/012017; (d) artikel sudah diaccepted untuk diterbitkan pada AIP Conference Proceeding terindeks scopus; (2) Artikel yang telah dipresentasikan pada International Conference on Advanced Materials Science and Technology (ICAMST 2017) sebanyak 5 artikel; (3) *Artikel submitted* pada Jurnal Internasional Materials Science and Engineering penerbit IOP publishing UK terindeks SJR dan Scopus sebanyak 5 artikel; (4) *Invited Speaker* pada *One Asia Convention* Nagoya 2017; (5) Paten status pemeriksaan substantif, judul: Paduan Oksida Logam $Nd_{1.2}Fe_1O_3$ dan Metode Pembuatannya, pada Direktorat Jenderal Hak Kekayaan Intelektual, Kementerian Hukum dan Hak Asasi Manusia, Republik Indonesia, Nomor Pendaftaran P00201703620, Tgl 8 Juni 2017; dan (6) satu draft buku ajar (*in review*) untuk penerbitan ISBN dengan judul Karakteristik Bahan Oksida.

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Penulis sepenuhnya menyadari bahwa laporan ini masih jauh dari kesempurnaan baik dari segi bahasa, sistematika penulisan maupun isi yang terkandung di dalamnya. Oleh karena itu penulis sangat mengharapkan kritikan dan saran yang sifatnya dapat membangun demi kesempurnaan penulisan laporan ini. Melalui kesempatan ini, tak lupa penulis ucapkan terima kasih kepada:

Akhirnya hanya kepada Allah SWT, penulis memohon ridha dan magfirahnya, Semoga segala dukungan serta bantuan semua pihak mendapat pahala yang berlipat ganda disisi Allah SWT, semoga karya ini dapat bermanfaat kepada para pembaca, Amin. Wassalam.

Makassar, 2017
Penulis,

TIM PENELITI

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