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We have completed the revision of the manuscript as appended. We also have sent the revised manuscript to a trusted colleague with considerable technical English writing and editing skills as a proofreader.

Crystal Structure of $Nd_{1.2}FeO_3$ Oxide Material and Its Rietveld Refinement Analysis

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Abstract

The crystal structure of $Nd_{1.2}FeO_3$ oxide material ~~has been~~ synthesized ~~with the variation~~ by varying ~~of~~ calcination temperature~~s~~ ~~and has characterized~~ was determined using the X-ray diffraction method. Further analysis by Rietveld refinement using software Rietica showed that all of the samples have an orthorhombic phase structure. The lattice constants of each a sample with variation of calcination temperature is a = 5.581059 ± 0.000736 Å, b = 7.758627 ± 0.000947 Å, c = 5.448341 ± 0.000665 Å; a = 5.580203 ± 0.000695 Å, b = 7.756789 ± 0.000908 Å, c = 5.447646 ± 0.000626 Å; and a = 5.580402 ± 0.000704 Å, b = 7.758957 ± 0.000919 Å, c = 5.449350 ± 0.000634 Å, respectively. The results of lattice constant were associated with the value of Goodness of Fit (GoF) is 0.9101%, 0.8726%, and 0.9303%, respectively. That has a strong indication of a qualified matching between the $NdFeO_3$ model numbers of COD 2003124 with the current experimental results. The value of FWHM and the crystal size of $Nd_{1.2}FeO_3$ samples are 0.22º and 372 nm. The results showed that the variation of calcination temperature has not a significant change in the crystal size and homogeneity of the atomic crystal structure. These results are confirmed by simulation of the atomic structure using the Diamond software, the dominant peak of *hkl* (121).
Keywords: X-ray diffraction; FWHM; $Nd_{1.2}FeO_3$; Rietveld refinement; Crystal Structure; Morphology.

1. Introduction

$NdFeO_3$ compounds which have a perovskite structure with the general formula of $RFeO_3$ (R = Rare-Earth) have investigated its utility in a wide variety of applications such as in solid oxide fuel cells [1], gas sensors [2, 3], the photo-catalysis and catalytic converters [4-6]. $NdFeO_3$ has a perovskite-type orthorhombic structure [7, 8]. In $NdFeO_3$ compounds, there are three main magnetic interactions: Fe-Fe, Nd-Fe, and Nd-Nd [9]. Such interaction competes in determining the structure and properties of attractive magnetic that trigger the number of applications. One of the applications of nanopowders $NdFeO_3$ is a gas sensor to detect H_2S [10] and C_2H_5OH [11]. $NdFeO_3$ oxide material has been successfully synthesized by using various methods, such as hydrothermal [12], combustion [13, 14], sol-gel [15], precipitation method [16] and sonication assisted precipitation [17]. The solid-state reaction is the conventional method is most often used for the synthesis of ceramic compounds [8, 18], in which the process is relatively cheap and easy, and the product of this reaction also has a high purity level and good crystalline compared to the other methods. Authors have experience in the fabrication of such an oxide material, e.g., $YBa_2Cu_3O_y$, NdBaCuO (off-stoichiometric), and $NdFe_xBa_{2-x}Cu_3O_y$, the results have reported elsewhere [19, 20, 21].

In this research, we reported our current results in the development of NdFeO oxide material as one potential candidate for sensor application. Further analysis is to determine quantitatively the physical characteristics of the material by X-ray diffraction data using Rietveld. Rietveld is a method of matching the theoretical curve with the experimental curve until there is an agreement between the two curves as a whole [22]. Based on this analysis, the crystal quality of materials can be concluded.

$NdFeO_3$ crystal structure is described in the space group Pbnm [23, 24]. Atom is located in crystallographic sites below: Nd_{3+} ion in (4c), Fe_{3+} in (4b) and ion O_2 (4c) and (8d). Fe_{3+} ions are coordinated by six ions O_2 that form octahedral FeO_6. The unit cell is composed of octahedral angle FeO_6 that in which the tilt angle as a function of temperature [25]. In this article, $Nd_{1.2}FeO_3$ synthesized using a solid-state reaction method with variations of calcination temperature. Characterization of material has been done by X-ray diffraction (XRD), which includes phase identification and quantitative analysis using Rietica software, Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS), includes fast EDS mapping.

2. Experimental Procedure

$Nd_{1+x}FeO_3$ oxide material has been grown by using the solid-state method with a variation of molar ratio, sintering temperature, and annealing temperature. The best results with a variety of sintering and annealing temperature are using a molar ratio of x = 0.2 [26]. This current research is reported of synthesis of $Nd_{1.2}FeO_3$ material using raw materials of Nd_2O_3 99.99% purchased by Strem Chemicals-USA and Fe_2O_3 99.99% purchased by Sigma-Aldrich-USA with calcined temperature was varied. Raw materials were mixed using a solid-state reaction method with the molar ratio of x = 0.2. The raw material powder Nd_2O_3 99.99% and Fe_2O_3 99.99% were weighed according to the stoichiometric calculations to get an oxide material $Nd_{1.2}FeO_3$. The mixture of $Nd_{1.2}FeO_3$ that obtained was grinded for 3 hours and calcined at a temperature of 700ºC for 6 hours, then crushed for 5 hours and sintering for 6 hours at a temperature of 950ºC. The synthesis process and the heating then repeated to obtain a better sample homogeneity [8, 27, 28, 29, 30]. After the samples grinded for 3 hours and repeat to calcinate with variations in temperature 750ºC, 850ºC, and 950ºC, respectively. The product of calcination powder and then grinded back for 5 hours and sintering at a temperature of 950ºC for 6 hours following by cooling down the process until reached of ambient room temperature.

$Nd_{1.2}FeO_3$ powder characterized by X-ray diffraction [Rigaku MiniFlex II, 2θ = 20º - 65º (CuKα, λ = 0.154 nm)] to determine the crystal structure and quantitatively analyzed by Rietveld refinement method using software Rietica. The parameters refined include (1) global parameters: sample displacement and Polynomial background of coefficient function (order of 5) and (2) the parameters phases: the lattice parameters, factor scale, component Gaussian (U), component Gamma (Gam0), asymmetry peak, and preferred orientation. Output parameters used to determine the results of refinement crystallinity and lattice parameters of the samples and the illustration of the atomic structure using the Diamond software.

The analysis of morphology and elemental of the $Nd_{1.2}FeO_3$ powder investigated using FEI Quanta FEG Scanning Electron Microscope (SEM) and Fast Energy Dispersive Spectroscopy (EDS) mapping with magnification 50,000x and 100,000x times.

3. Results and Discussion

3.1 X-Ray Diffraction Analysis

XRD diffraction patterns of oxide material $Nd_{1.2}FeO_3$ powder were synthesized by using the solid-state reaction method with variations of calcination temperature at a temperature of 750ºC, 850ºC, and 950ºC, as shown in Figure 1.

Figure 1 shows the peak of $Nd_{1.2}FeO_3$, and the Nd_2O_3 phase has been identified based on the adjustment of the data using software Match!. This crystallographic curve indicates that the raw materials of Nd_2O_3 and Fe_2O_3 have formed a new $NdFeO_3$ phase. The existence of the formation of a minor Nd_2O_3 phase is an indication that the raw materials are not entirely produced in the $Nd_{1.2}FeO_3$ phase. Imperfection reaction suspected due to the adjustment of calcination temperature, and the heating time is less than optimal. This, according to another study, has explained that the $NdFeO_3$ single phase could be formed with a calcination temperature of 1000ºC and sintered at a temperature of 1200ºC. However, the results obtain defects and also contained cracks [31].

Another researcher, Niu Xinshu et al., also succeeds in synthesized $NdFeO_3$ with a temperature of 950ºC [9] and Yabin Wang et al. with a temperature of 1000ºC [31]. Their results were similar to the current research with the indication of forming the $NdFeO_3$ dominant phase located at 2θ = 32.56º associated with *hkl* value (121). The intensity of the dominant phase of *hkl* (121) increases if the heating temperature is increased [32].

Calculating the crystal size can be estimated by using Debye-Scherer equation as described in Equation (1)

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

Where λ is the wavelength of the radiation Cu Kα (λ = 0,154 nm), θ is the angle Bragg (º), and β = FWHM at the peak of *hkl* (121) is association 2θ of 32.56º [27]. The calculation results of crystal size and FWHM can be seen in Table 1.

Figure 2 presented the calculation result of the relative intensities as a variation of calcination temperature related to the value of FWHM. The crystal size and homogeneity of surface morphology is no significant change. The existence of the atom due to the Nd_2O_3 phase will reduce the diffraction intensity of each sample. However, the sample with a calcination temperature of 850ºC is shown

more dominant at the peak of *hkl* (121). In contrast, it can be seen that the FWHM values for each sample are the same in order of 0.22º. Therefore, the $Nd_{1.2}FeO_3$ oxide material with the dominant peak of hkl 121 and the parameter process, as has explained above, will be useful for the application as gas sensors as has reported elsewhere [2,3,10,11] for the fast detection of ethanol, propylene, NO_2, CO, C_3H_8, and C_6H_{14}. This result is similar, as has reported [33], FWHM for each peak in the orthorhombic phase has smaller with increasing heating temperature.

3.2 Rietveld Refinement Analysis

In this research, models made from the data COD corresponding to the materials used, for a Nd_2O_3 number of COD 2002849 [34], while for a $NdFeO_3$ number of COD 2003124 [23].

Figures 3, 4, and 5 show a plot of the results of Rietveld refinement data diffraction of three samples powder $Nd_{1.2}FeO_3$ with the angle of 2θ of 10-70º. The measurable pattern indicated by the sign (+), the calculated pattern shown by the red line, whereas the green line shows the difference between both of them. Suitability (figures-of-merit) of refinement shaws in Table 2. The three images formed that the fit between the calculated data and measured data is quite well. There are no other peak differences and plot the difference is not fluctuated significantly, indicating that the Rietveld refinement acceptable under the required criteria, which is the GoF <4% and RWP <20% as was reported Kisi [35]. Other researchers reported compounds $NdFeO_3$ calcined at temperatures of 750ºC to obtain orthorhombic structure (Pnma) by using the sol-gel route technique, with refinement parameter Rietveld Rp = 8.6, RWP = 6.1, and Goodness of Fit (GoF) = 1.6 [36].

The output of Rietveld refinement analysis showed that the crystal structure of the material of $Nd_{1.2}FeO_3$ is orthorhombic with space group is Pnma, $a \neq b \neq c$, $\alpha = \beta = \gamma = 90°$ to the respective lattice parameters are presented in Table 3. This result is comparable with has reported other researchers ~~was~~ that synthesize $NdFeO_3$ using sol-gel method citrate obtained $NdFeO_3$ phase with orthorhombic structure (Pnma) with the lattice parameters is a = 5.578 Å, b = 7.758 Å, c = 5.448 Å [32].

Table 4 is the weight percentage of each phase, the highest weight percentage obtained at the YK950 sample of 99.46%. The increase of calcination temperature tends to weight the percentage of $Nd_{1.2}FeO_3$ phase increased and decreased of Nd_2O_3 phase. This shows that the ions Nd_{+3} and Fe_{+3} forms of $NdFeO_3$ oxide material were improving without any impurity with increasing calcination temperature.

The results of structure visualization of $Nd_{1.2}FeO_3$ unit's cell based on lattice parameters have obtained from Rietveld refinement analysis described using software DIAMOND, as shown in Figures 6, 7, and 8, respectively. The orthorhombic crystal structure with the space group Pnma, in which grey spheres illustrate cation Nd_{+3}, green spheres represent cation Fe_{+3}, and the red spheres are oxygen ions with the Wyckoff of position of each ion Nd_{+3} on (4c), ion Fe_{+3} in (4b) and ion O_2 (4c) and (8d). All lattice parameters *a, b,* and *c* are similar to each sample variation.

3.3 Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) Analysis

SEM ~~has~~ was used as a method for analyzing the surface morphology of the $Nd_{1.2}FeO_3$ sample shown in figures 9 and 10 with magnifications of 50,000x, and 100,000x, it can be seen that all samples have high homogeneity indicated by the morphology of the sample forming small uniform granules. These similar results also reported by Sujiono et al. [28]. The $Nd_{1.2}FeO_3$ sample, which is calcined at 750°C, has a grain size 25.63 nm (Figures 9a) and the $Nd_{1.2}FeO_3$ sample, which is calcined at 950°C has a grain size ranging from 14.16 nm – 22.93 nm (Figures 10b). The difference in grain size between the two samples is due to the calcination temperature. On the other terms of this powder has a high porosity so that it becomes one of the advantages to improve its characteristics as a gas sensor [3].

Furthermore, the EDS, it has confirmed that consistently form Nd, Fe, O with an average stoichiometric ratio of 1.2: 1: 3 which contained Nd (53.90 wt%), Fe (26.49 wt%), O (15.09 wt%), C (4.52 wt%), and Nd (49.81 wt%), Fe (23.23 wt%), O (16.80 wt%), C (10.16 wt%) respectively as shown in table 5.

Elemental maps of the $Nd_{1.2}FeO_3$ samples surface area are shown in Figures 11 and 12, respectively. Figures 11a and 12a contain a secondary electron (SE) image of the $Nd_{1.2}FeO_3$ samples as well as the corresponding maps of the distribution of chemical elements on the scanned surface. The elemental mapping of the $Nd_{1.2}FeO_3$ samples shows that the surface area was rich with neodymium (in neodymium-rich area, see Nd-MAB map), iron (in iron-rich area, see Fe-K map), and oxygen (see O-K map), respectively. The presence of carbon in EDS results is origin from carbon

adhesive tape. There are no impurities seen, and it can be confirmed that the results of the XRD and SEM-EDS analyzes were consistent with the dominant peak of hkl 121 [10,11].

4. Conclusion

$Nd_{1.2}FeO_3$ oxide material has successfully synthesized as a basis of the material gas sensor. The results of X-ray diffraction analysis showed $NdFeO_3$ and Nd_2O_3 phase, in which the crystal structure of the phase $Nd_{1.2}FeO_3$ is orthorhombic to the space group Pnma. $NdFeO_3$ phase lattice constant for each sample is YK750 a = 5.581059 ± 0.000736 Å, b = 7.758627 ± 0.000947 Å, c = 5.448341 ± 0.000665 Å; YK850 a = 5.580203 ± 0.000695 Å, b = 7.756789 ± 0.000908 Å, c = 5.447646 ± 0.000626 Å; and YK950 a = 5.580402 ± 0.000704 Å, b = 7.758957 ± 0.000919 Å, c = 5.449350 ± 0.000634 Å, respectively related to the GoF values <1% and the estimated size of the crystals is 372 nm. In the analysis of surface area data and elemental compositions confirmed that $Nd_{1.2}FeO_3$ samples have homogeneous morphology and grain sizes that tend to be uniform.

Acknowledgments

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The revised submission has addressed all the editor's comments in full.
1. We added supplementary characterize the materials by SEM/EDAX and have included the results as part of the properties of materials.
2. We agree with the editors and have revised the Results and Discussion section.
This research has not studied the response of the materials to the specific a gas but investigated the relation of the parameters process to the crystal structure and surface morphology. Based on the characteristics and the data, then analyzed using Rietica and simulated using Diamond software. The obtained dominant peak of hkl 121, which is sensitive to various types of gas have described in detail and include the reference in the revised manuscript.

Thank you very much for your consideration.

Yours Sincerely,
Prof. Eko Hadi Sujiono
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> Authors designed $Nd_{1.2}FeO_3$ oxide materials and performed a detailed structural characterization. However, some aspects still need to be further elucidated. Therefore, I can't recommend this manuscript to publish in the "Romanian Journal of Materials" in the present form.
> Specific comments:
>
> 1 The prepared materials are not fully investigated. XRD analysis and Rietveld refinement were employed for Nd1.2FeO3 oxide material characterization in this manuscript, but it is insufficient. The authors should supplementary characterize the materials by different techniques, for example, SEM/EDAX knowing that the morphology has a significant influence on the properties of materials.
> 2. Furthermore, the response of the materials as a gas sensor should be investigated. The authors stated in the conclusion part that: "This study has obtained Nd1.2FeO3 oxide material with the dominant peak of hkl 121 which is sensitive to various types of gas as has been reported elsewhere". It seems to me someone has reported this? If so, please give details and include the reference. The sentence should be moved to the results and discussion section.
> 3. The authors should check the manuscript carefully to avoid grammar mistakes.

În mie., 13 nov. 2019 la 03:53, Prof. Dr. Eko Hadi Sujiono, M.Si UNM <e.h.sujiono@unm.ac.id> a scris:

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I am submitting a manuscript for consideration of publication in the Romanian Journal of Materials. The article is entitled "Crystal Structure of $Nd_{1.2}FeO_3$ Oxide Material and Its Rietveld Refinement Analysis for Gas Sensor Application".

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Crystal Structure of $Nd_{1.2}FeO_3$ Oxide Material and Its Rietveld Refinement Analysis

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Abstract

The crystal structure of $Nd_{1.2}FeO_3$ oxide material has been synthesized with the variation of calcination temperature and has characterized using the X-ray diffraction method. Further analysis by Rietveld refinement using software Rietica showed that all of the samples have an orthorhombic phase structure. The lattice constants of each a sample with variation of calcination temperature is a = 5.581059 ± 0.000736 Å, b = 7.758627 ± 0.000947 Å, c = 5.448341 ± 0.000665 Å; a = 5.580203 ± 0.000695 Å, b = 7.756789 ± 0.000908 Å, c = 5.447646 ± 0.000626 Å; and a = 5.580402 ± 0.000704 Å, b = 7.758957 ± 0.000919 Å, c = 5.449350 ± 0.000634 Å, respectively. The results of lattice constant were associated with the value of Goodness of Fit (GoF) is 0.9101%, 0.8726%, and 0.9303%, respectively. That has a strong indication of a qualified matching between the $NdFeO_3$ model numbers of COD 2003124 with the current experimental results. The value of FWHM and the crystal size of $Nd_{1.2}FeO_3$ samples are 0.22º and 372 nm. The results showed that the variation of calcination temperature has not a significant change in the crystal size and homogeneity of the atomic crystal structure. These results are confirmed by simulation of the atomic structure using the Diamond software, the dominant peak of *hkl* (121).
Keywords: X-ray diffraction; FWHM; $Nd_{1.2}FeO_3$; Rietveld refinement; Crystal Structure; Morphology.

1. Introduction

$NdFeO_3$ compounds which have a perovskite structure with the general formula of $RFeO_3$ (R = Rare-Earth) have investigated its utility in a wide variety of applications such as in solid oxide fuel cells [1], gas sensors [2, 3], the photo-catalysis and catalytic converters [4-6]. $NdFeO_3$ has a perovskite-type orthorhombic structure [7, 8]. In $NdFeO_3$ compounds, there are three main magnetic interactions: Fe-Fe, Nd-Fe, and Nd-Nd [9]. Such interaction competes in determining the structure and properties of attractive magnetic that trigger the number of applications. One of the applications of nanopowders $NdFeO_3$ is a gas sensor to detect H_2S [10] and C_2H_5OH [11]. $NdFeO_3$ oxide material has been successfully synthesized by using various methods, such as hydrothermal [12], combustion [13, 14], sol-gel [15], precipitation method [16] and sonication assisted precipitation [17]. The solid-state reaction is the conventional method is most often used for the synthesis of ceramic compounds [8, 18], in which the process is relatively cheap and easy, and the product of this reaction also has a high purity level and good crystalline compared to the other methods. Authors have experience in the fabrication of such an oxide material, e.g., $YBa_2Cu_3O_y$, NdBaCuO (off-stoichiometric), and $NdFe_xBa_{2-x}Cu_3O_y$, the results have reported elsewhere [19, 20, 21].

In this research, we reported our current results in the development of NdFeO oxide material as one potential candidate for sensor application. Further analysis is to determine quantitatively the physical characteristics of the material by X-ray diffraction data using Rietveld. Rietveld is a method of matching the theoretical curve with the experimental curve until there is an agreement between the two curves as a whole [22]. Based on this analysis, the crystal quality of materials can be concluded.

$NdFeO_3$ crystal structure is described in the space group Pbnm [23, 24]. Atom is located in crystallographic sites below: Nd_{3+} ion in (4c), Fe_{3+} in (4b) and ion O_2 (4c) and (8d). Fe_{3+} ions are coordinated by six ions O_2 that form octahedral FeO_6. The unit cell is composed of octahedral angle FeO_6 that in which the tilt angle as a function of temperature [25]. In this article, $Nd_{1.2}FeO_3$ synthesized using a solid-state reaction method with variations of calcination temperature. Characterization of material has been done by X-ray diffraction (XRD), which includes phase identification and quantitative analysis using Rietica software, Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS), includes fast EDS mapping.

2. Experimental Procedure

$Nd_{1+x}FeO_3$ oxide material has been grown by using the solid-state method with a variation of molar ratio, sintering temperature, and annealing temperature. The best results with a variety of sintering and annealing temperature are using a molar ratio of x = 0.2 [26]. This current research is reported of synthesis of $Nd_{1.2}FeO_3$ material using raw materials of Nd_2O_3 99.99% purchased by Strem Chemicals-USA and Fe_2O_3 99.99% purchased by Sigma-Aldrich-USA with calcined temperature was varied. Raw materials were mixed using a solid-state reaction method with the molar ratio of x = 0.2. The raw material powder Nd_2O_3 99.99% and Fe_2O_3 99.99% were weighed according to the stoichiometric calculations to get an oxide material $Nd_{1.2}FeO_3$. The mixture of $Nd_{1.2}FeO_3$ that obtained was grinded for 3 hours and calcined at a temperature of 700ºC for 6 hours, then crushed for 5 hours and sintering for 6 hours at a temperature of 950ºC. The synthesis process and the heating then repeated to obtain a better sample homogeneity [8, 27, 28, 29, 30]. After the samples grinded for 3 hours and repeat to calcinate with variations in temperature 750ºC, 850ºC, and 950ºC, respectively. The product of calcination powder and then grinded back for 5 hours and sintering at a temperature of 950ºC for 6 hours following by cooling down the process until reached of ambient room temperature.

$Nd_{1.2}FeO_3$ powder characterized by X-ray diffraction [Rigaku MiniFlex II, 2θ = 20º - 65º (CuKα, λ = 0.154 nm)] to determine the crystal structure and quantitatively analyzed by Rietveld refinement method using software Rietica. The parameters refined include (1) global parameters: sample displacement and Polynomial background of coefficient function (order of 5) and (2) the parameters phases: the lattice parameters, factor scale, component Gaussian (U), component Gamma (Gam0), asymmetry peak, and preferred orientation. Output parameters used to determine the results of refinement crystallinity and lattice parameters of the samples and the illustration of the atomic structure using the Diamond software.

The analysis of morphology and elemental of the $Nd_{1.2}FeO_3$ powder investigated using FEI Quanta FEG Scanning Electron Microscope (SEM) and Fast Energy Dispersive Spectroscopy (EDS) mapping with magnification 50,000x and 100,000x times.

3. Results and Discussion

3.1 X-Ray Diffraction Analysis

XRD diffraction patterns of oxide material $Nd_{1.2}FeO_3$ powder were synthesized by using the solid-state reaction method with variations of calcination temperature at a temperature of 750ºC, 850ºC, and 950ºC, as shown in Figure 1.

Figure 1 shows the peak of $Nd_{1.2}FeO_3$, and the Nd_2O_3 phase has been identified based on the adjustment of the data using software Match!. This crystallographic curve indicates that the raw materials of Nd_2O_3 and Fe_2O_3 have formed a new $NdFeO_3$ phase. The existence of the formation of a minor Nd_2O_3 phase is an indication that the raw materials are not entirely produced in the $Nd_{1.2}FeO_3$ phase. Imperfection reaction suspected due to the adjustment of calcination temperature, and the heating time is less than optimal. This, according to another study, has explained that the $NdFeO_3$ single phase could be formed with a calcination temperature of 1000ºC and sintered at a temperature of 1200ºC. However, the results obtain defects and also contained cracks [31].

Another researcher, Niu Xinshu et al., also succeeds in synthesized $NdFeO_3$ with a temperature of 950ºC [9] and Yabin Wang et al. with a temperature of 1000ºC [31]. Their results were similar to the current research with the indication of forming the $NdFeO_3$ dominant phase located at 2θ = 32.56º associated with *hkl* value (121). The intensity of the dominant phase of *hkl* (121) increases if the heating temperature is increased [32].

Calculating the crystal size can be estimated by using Debye-Scherer equation as described in Equation (1)

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

Where λ is the wavelength of the radiation Cu Kα (λ = 0,154 nm), θ is the angle Bragg (º), and β = FWHM at the peak of *hkl* (121) is association 2θ of 32.56º [27]. The calculation results of crystal size and FWHM can be seen in Table 1.

Figure 2 presented the calculation result of the relative intensities as a variation of calcination temperature related to the value of FWHM. The crystal size and homogeneity of surface morphology is no significant change. The existence of the atom due to the Nd_2O_3 phase will reduce the diffraction intensity of each sample. However, the sample with a calcination temperature of 850ºC is shown

more dominant at the peak of *hkl* (121). In contrast, it can be seen that the FWHM values for each sample are the same in order of 0.22º. Therefore, the $Nd_{1.2}FeO_3$ oxide material with the dominant peak of hkl 121 and the parameter process, as has explained above, will be useful for the application as gas sensors as has reported elsewhere [2,3,10,11] for the fast detection of ethanol, propylene, NO_2, CO, C_3H_8, and C_6H_{14}. This result is similar, as has reported [33], FWHM for each peak in the orthorhombic phase has smaller with increasing heating temperature.

3.2 Rietveld Refinement Analysis

In this research, models made from the data COD corresponding to the materials used, for a Nd_2O_3 number of COD 2002849 [34], while for a $NdFeO_3$ number of COD 2003124 [23].

Figures 3, 4, and 5 show a plot of the results of Rietveld refinement data diffraction of three samples powder $Nd_{1.2}FeO_3$ with the angle of 2θ of 10-70º. The measurable pattern indicated by the sign (+), the calculated pattern shown by the red line, whereas the green line shows the difference between both of them. Suitability (figures-of-merit) of refinement shaws in Table 2. The three images formed that the fit between the calculated data and measured data is quite well. There are no other peak differences and plot the difference is not fluctuated significantly, indicating that the Rietveld refinement acceptable under the required criteria, which is the GoF <4% and RWP <20% as was reported Kisi [35]. Other researchers reported compounds $NdFeO_3$ calcined at temperatures of 750ºC to obtain orthorhombic structure (Pnma) by using the sol-gel route technique, with refinement parameter Rietveld Rp = 8.6, RWP = 6.1, and Goodness of Fit (GoF) = 1.6 [36].

The output of Rietveld refinement analysis showed that the crystal structure of the material of $Nd_{1.2}FeO_3$ is orthorhombic with space group is Pnma, $a \neq b \neq c$, $\alpha = \beta = \gamma = 90°$ to the respective lattice parameters are presented in Table 3. This result is comparable with has reported other researchers was synthesize $NdFeO_3$ using sol-gel method citrate obtained $NdFeO_3$ phase with orthorhombic structure (Pnma) with the lattice parameters is a = 5.578 Å, b = 7.758 Å, c = 5.448 Å [32].

Table 4 is the weight percentage of each phase, the highest weight percentage obtained at the YK950 sample of 99.46%. The increase of calcination temperature tends to weight the percentage of $Nd_{1.2}FeO_3$ phase increased and decreased of Nd_2O_3 phase. This shows that the ions Nd_{+3} and Fe_{+3} forms of $NdFeO_3$ oxide material were improving without any impurity with increasing calcination temperature.

The results of structure visualization of $Nd_{1.2}FeO_3$ unit's cell based on lattice parameters have obtained from Rietveld refinement analysis described using software DIAMOND, as shown in Figures 6, 7, and 8, respectively. The orthorhombic crystal structure with the space group Pnma, in which grey spheres illustrate cation Nd_{+3}, green spheres represent cation Fe_{+3}, and the red spheres are oxygen ions with the Wyckoff of position of each ion Nd_{+3} on (4c), ion Fe_{+3} in (4b) and ion O_2 (4c) and (8d). All lattice parameters *a, b,* and *c* are similar to each sample variation.

3.3 Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) Analysis

SEM has used as a method for analyzing the surface morphology of the $Nd_{1.2}FeO_3$ sample shown in figures 9 and 10 with magnifications of 50,000x, and 100,000x, it can be seen that all samples have high homogeneity indicated by the morphology of the sample forming small uniform granules. These similar results also reported by Sujiono et al. [28]. The $Nd_{1.2}FeO_3$ sample, which is calcined at 750°C, has a grain size 25.63 nm (Figures 9a) and the $Nd_{1.2}FeO_3$ sample, which is calcined at 950°C has a grain size ranging from 14.16 nm – 22.93 nm (Figures 10b). The difference in grain size between the two samples is due to the calcination temperature. On the other terms of this powder has a high porosity so that it becomes one of the advantages to improve its characteristics as a gas sensor [3].

Furthermore, the EDS, it has confirmed that consistently form Nd, Fe, O with an average stoichiometric ratio of 1.2: 1: 3 which contained Nd (53.90 wt%), Fe (26.49 wt%), O (15.09 wt%), C (4.52 wt%), and Nd (49.81 wt%), Fe (23.23 wt%), O (16.80 wt%), C (10.16 wt%) respectively as shown in table 5.

Elemental maps of the $Nd_{1.2}FeO_3$ samples surface area are shown in Figures 11 and 12, respectively. Figures 11a and 12a contain a secondary electron (SE) image of the $Nd_{1.2}FeO_3$ samples as well as the corresponding maps of the distribution of chemical elements on the scanned surface. The elemental mapping of the $Nd_{1.2}FeO_3$ samples shows that the surface area was rich with neodymium (in neodymium-rich area, see Nd-MAB map), iron (in iron-rich area, see Fe-K map), and oxygen (see O-K map), respectively. The presence of carbon in EDS results is origin from carbon

adhesive tape. There are no impurities seen, and it can be confirmed that the results of the XRD and SEM-EDS analyzes were consistent with the dominant peak of hkl 121 [10,11].

4. Conclusion

$Nd_{1.2}FeO_3$ oxide material has successfully synthesized as a basis of the material gas sensor. The results of X-ray diffraction analysis showed $NdFeO_3$ and Nd_2O_3 phase, in which the crystal structure of the phase $Nd_{1.2}FeO_3$ is orthorhombic to the space group Pnma. $NdFeO_3$ phase lattice constant for each sample is YK750 a = 5.581059 ± 0.000736 Å, b = 7.758627 ± 0.000947 Å, c = 5.448341 ± 0.000665 Å; YK850 a = 5.580203 ± 0.000695 Å, b = 7.756789 ± 0.000908 Å, c = 5.447646 ± 0.000626 Å; and YK950 a = 5.580402 ± 0.000704 Å, b = 7.758957 ± 0.000919 Å, c = 5.449350 ± 0.000634 Å, respectively related to the GoF values <1% and the estimated size of the crystals is 372 nm. In the analysis of surface area data and elemental compositions confirmed that $Nd_{1.2}FeO_3$ samples have homogeneous morphology and grain sizes that tend to be uniform.

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Figure Captions

Fig. 1 - XRD pattern of $Nd_{1.2}FeO_3$ as variation of calcination temperature (♦ = $NdFeO_3$, • = Nd_2O_3).

Fig. 2 - The comparison of relative peak intensity and FWHM for both phases from three samples $Nd_{1.2}FeO_3$ with the variations of calcination temperature.

Fig. 3 - The plot of the Rietveld refinement results by using Rietica for sample YK750 oxide material $Nd_{1.2}FeO_3$ in the range 2θ of 10-70º.

Fig. 4 - The plot of the results Rietveld refinement by using Rietica for sample YK850 oxide material $Nd_{1.2}FeO_3$ in the range 2θ of 10-70º.

Fig. 5 - The plot of the results Rietveld refinement by using Rietica for sample YK950 oxide material $Nd_{1.2}FeO_3$ in the range 2θ of 10-70º.

Fig. 6 - Visualization atomic structure of $NdFe_{1.2}O_3$ phase calcined at temperatures of 750ºC to the lattice parameter is a = 5.581059 ± 0.000736 Å, b = 7.758627 ± 0.000947 Å and c = 5.448341 ± 0.000665 Å.

Fig. 7 - Visualization atomic structure of $NdFe_{1.2}O_3$ phase calcined at temperatures of 850ºC to the lattice parameter is a = 5.580203 ± 0.000695 Å, b = 7.756789 ± 0.000908 Å and c = 5.447646 ± 0.000626 Å.

Fig. 8 - Visualization atomic structure of $NdFe_{1.2}O_3$ phase calcined at temperatures of 950ºC to the lattice parameter is a = 5.580402 ± 0.000704 Å, b = 7.758957 ± 0.000919 Å and c = 5.449350 ± 0.000634 Å.

Fig. 9 - Morphology of sample $Nd_{1.2}FeO_3$ as a variation of calcination temperature YK750: a) Magnification 50,000x; b) Magnification 100,000x

Fig. 10 - Morphology of sample $Nd_{1.2}FeO_3$ as a variation of calcination temperature YK950: a) Magnification 50,000x; b) Magnification 100,000x

Fig. 11 – Analysis fast mapping EDS of sample $Nd_{1.2}FeO_3$: a) SE Image, b) EDS mapping of carbon, c) neodymium, d) iron, e) oxygen, and f) EDS mapping for all elements combined as a variation of calcination temperature YK750

Fig. 12 - Analysis fast mapping EDS of sample $Nd_{1.2}FeO_3$: a) SE Image, b) EDS mapping of carbon, c) neodymium, d) iron, e) oxygen, and f) EDS mapping for all elements combined as a variation of calcination temperature YK950

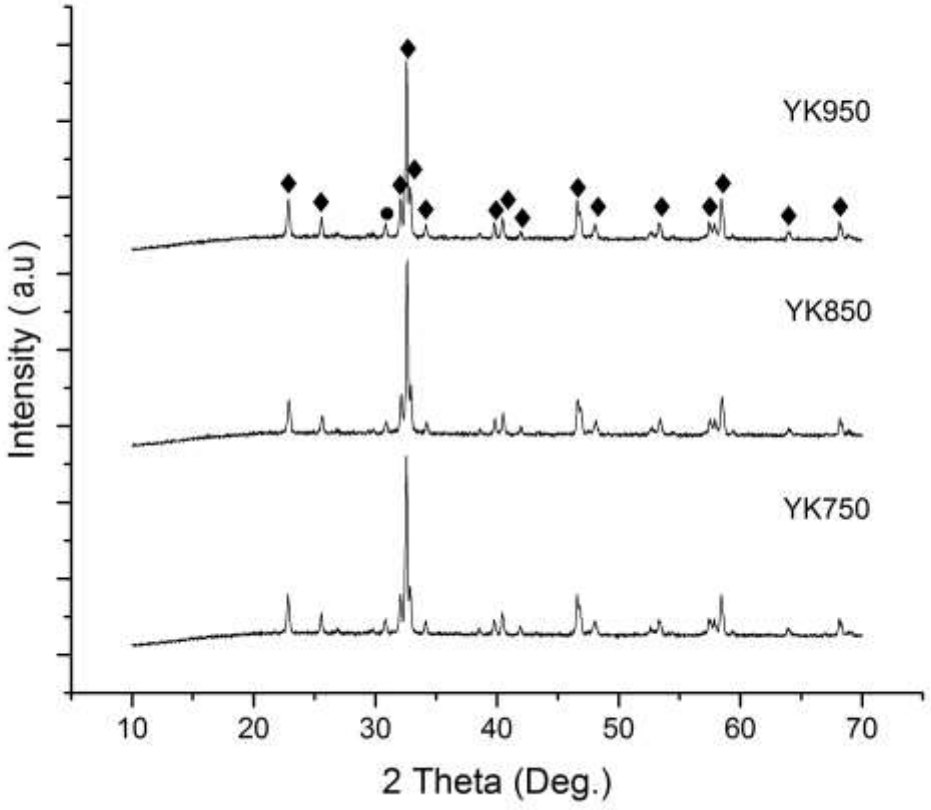


Fig. 1

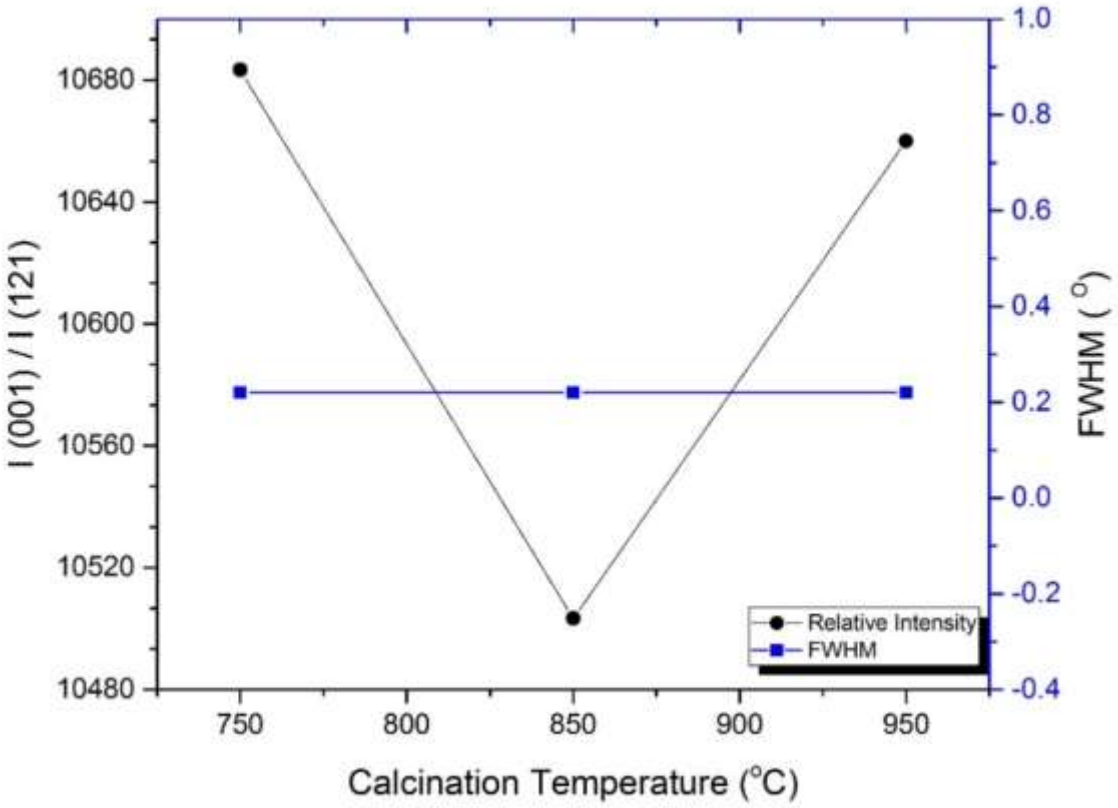
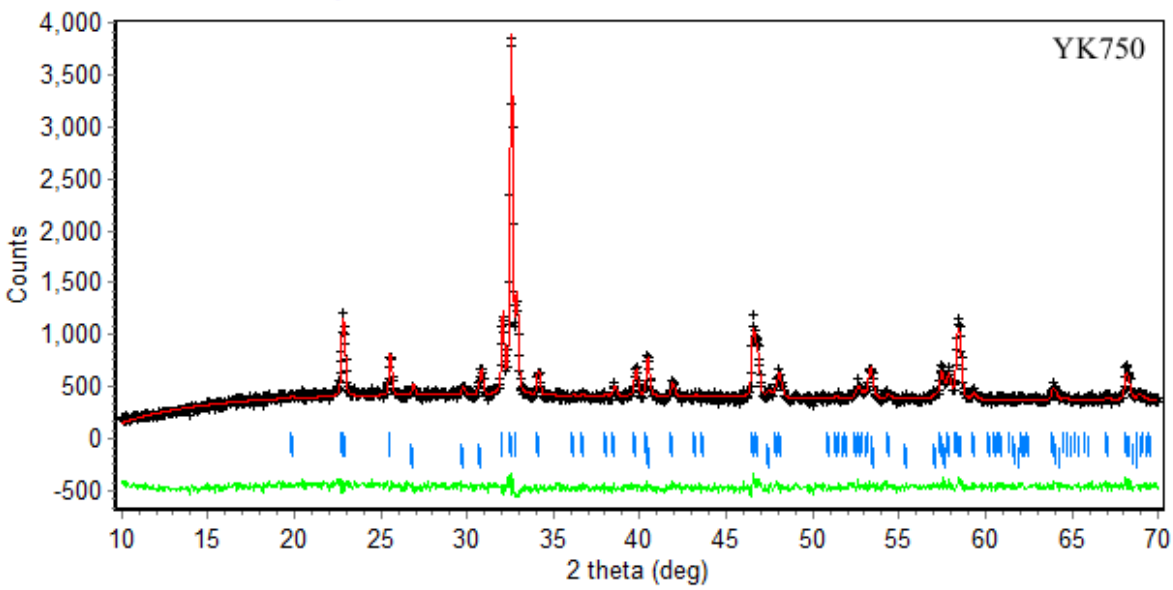

I (001) / I (121)
10680
10640
10600
10560
10520
10480
750
800
850
900
950
Calcination Temperature (°C)
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
-0.4
FWHM (°)
Relative Intensity
FWHM

Fig. 2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3,500
3,000
2,500
2,000
1,500
1,000
500
0
-500
10
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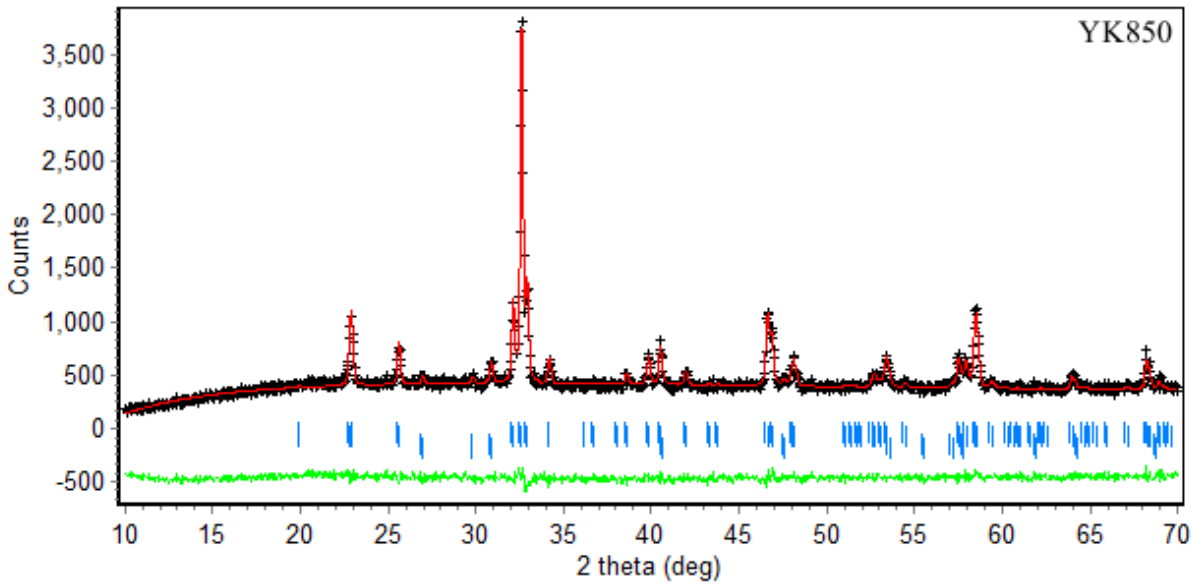
45
50
55
60
65
70
2 theta (deg)

Fig. 3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2,000
1,500
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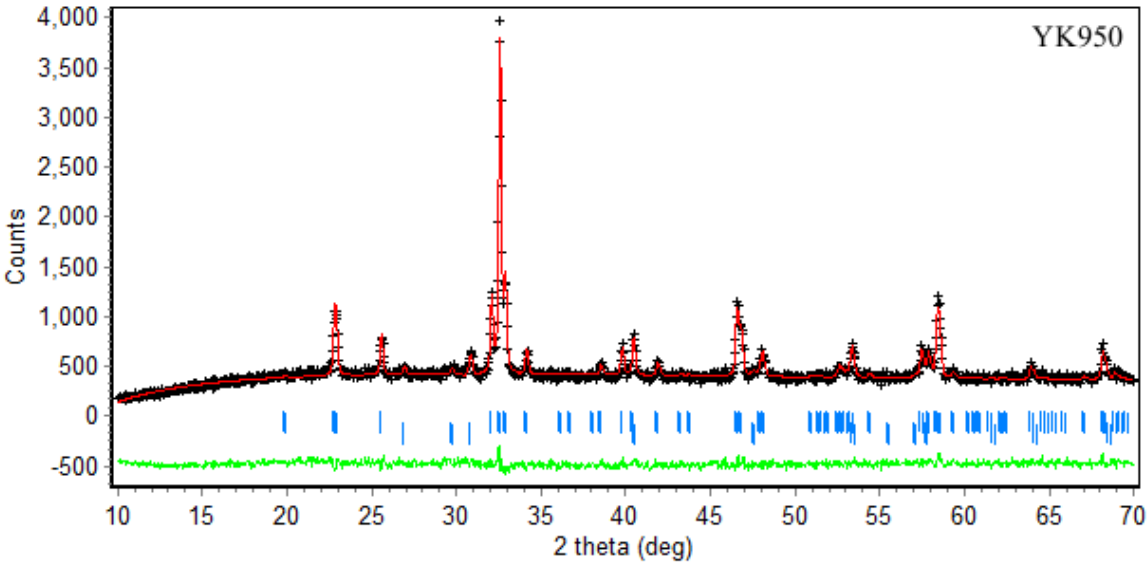
45
50
55
60
65
70
2 theta (deg)

Fig. 4


YK950
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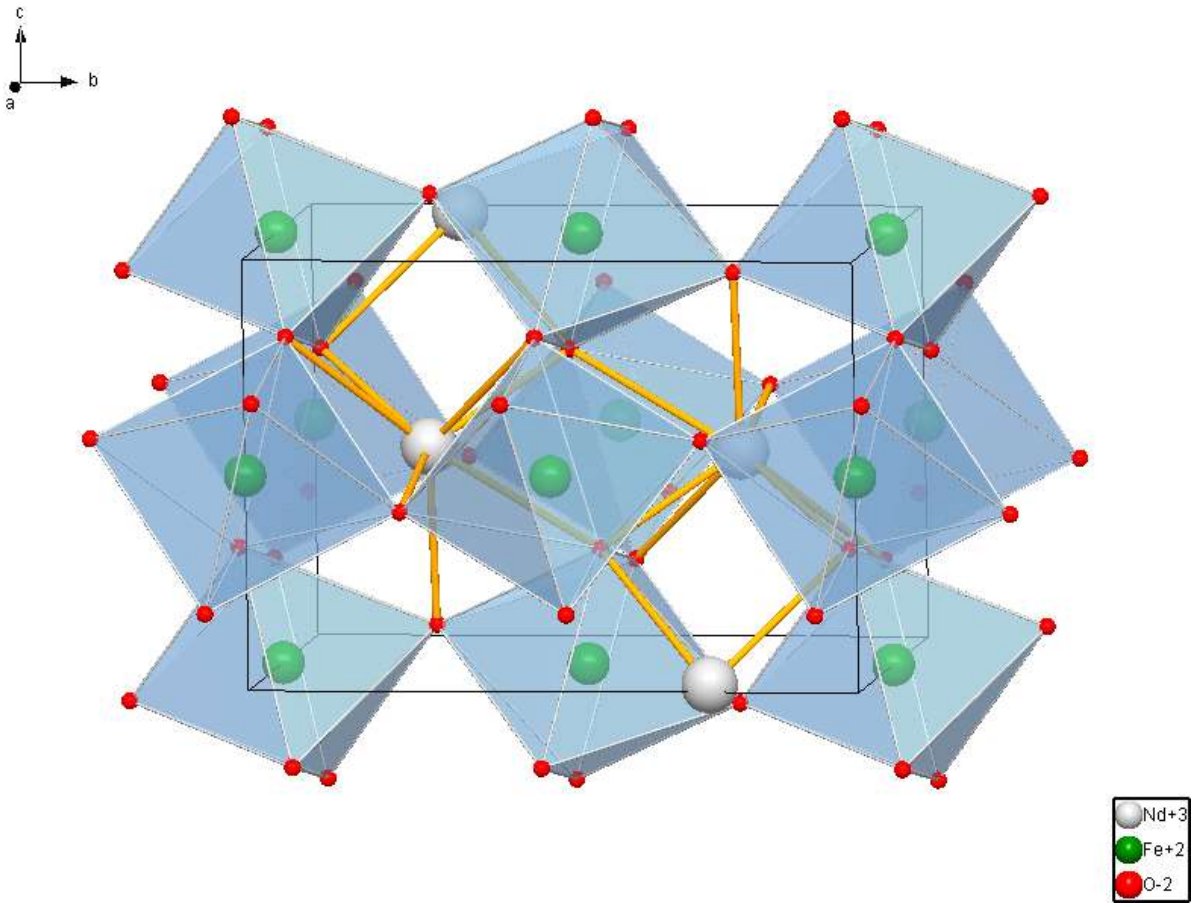
65
70
2 theta (deg)

Fig. 5



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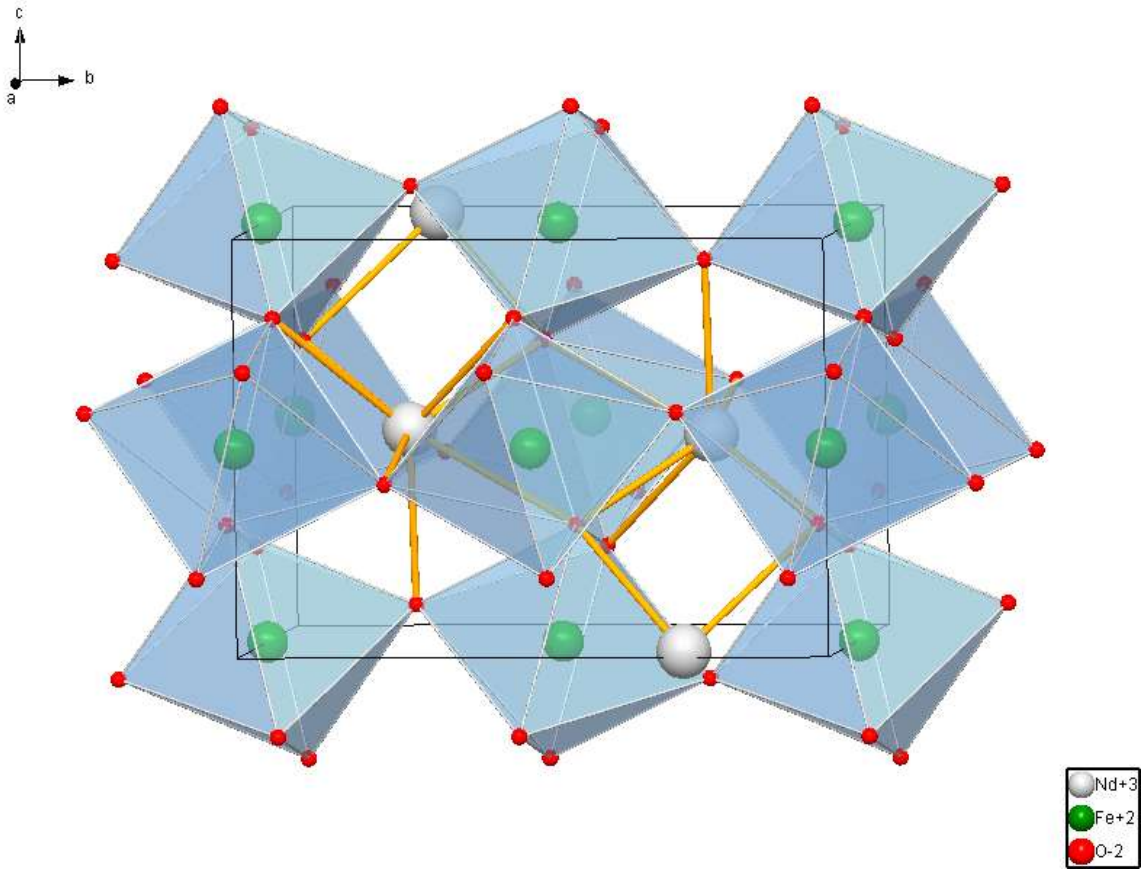

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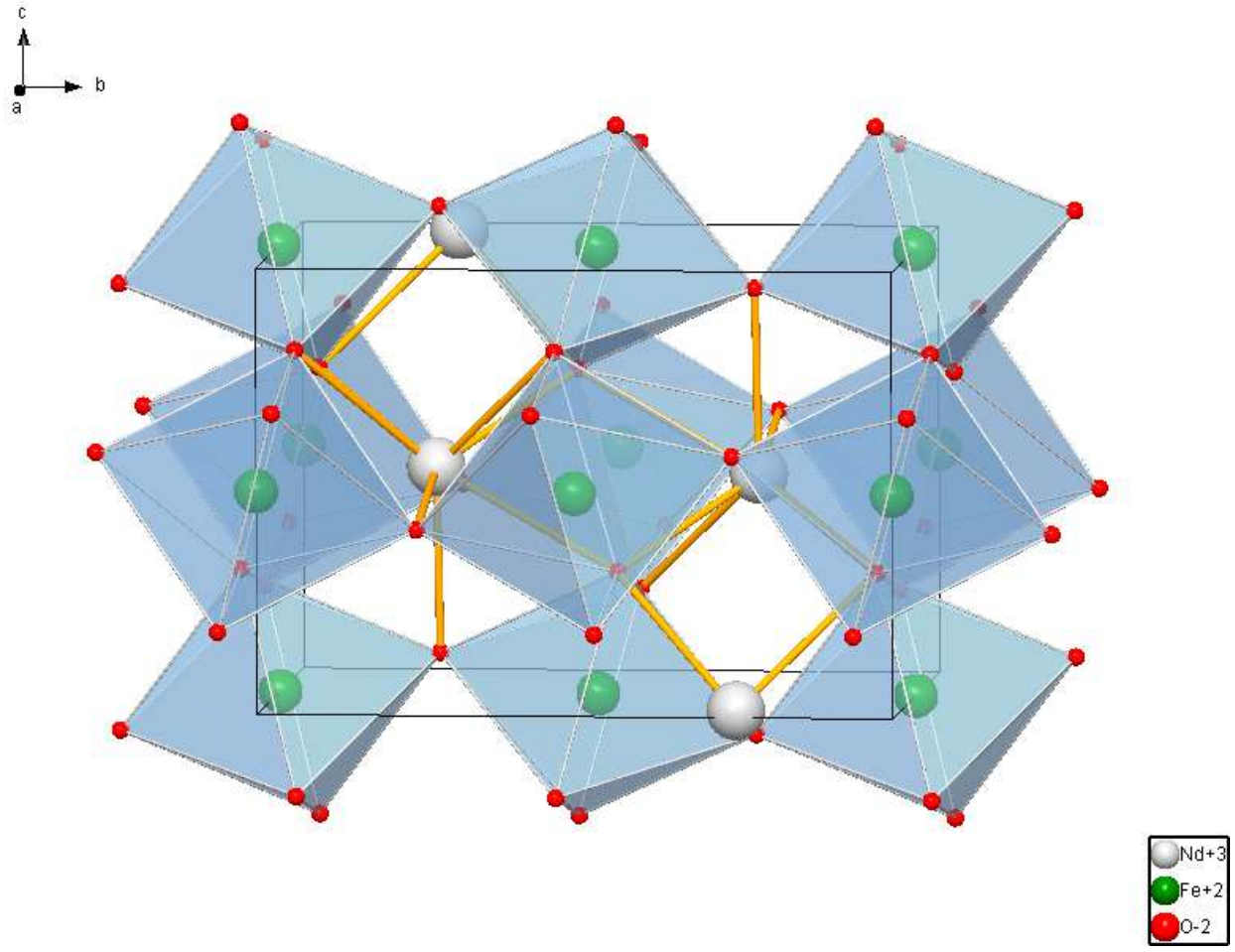

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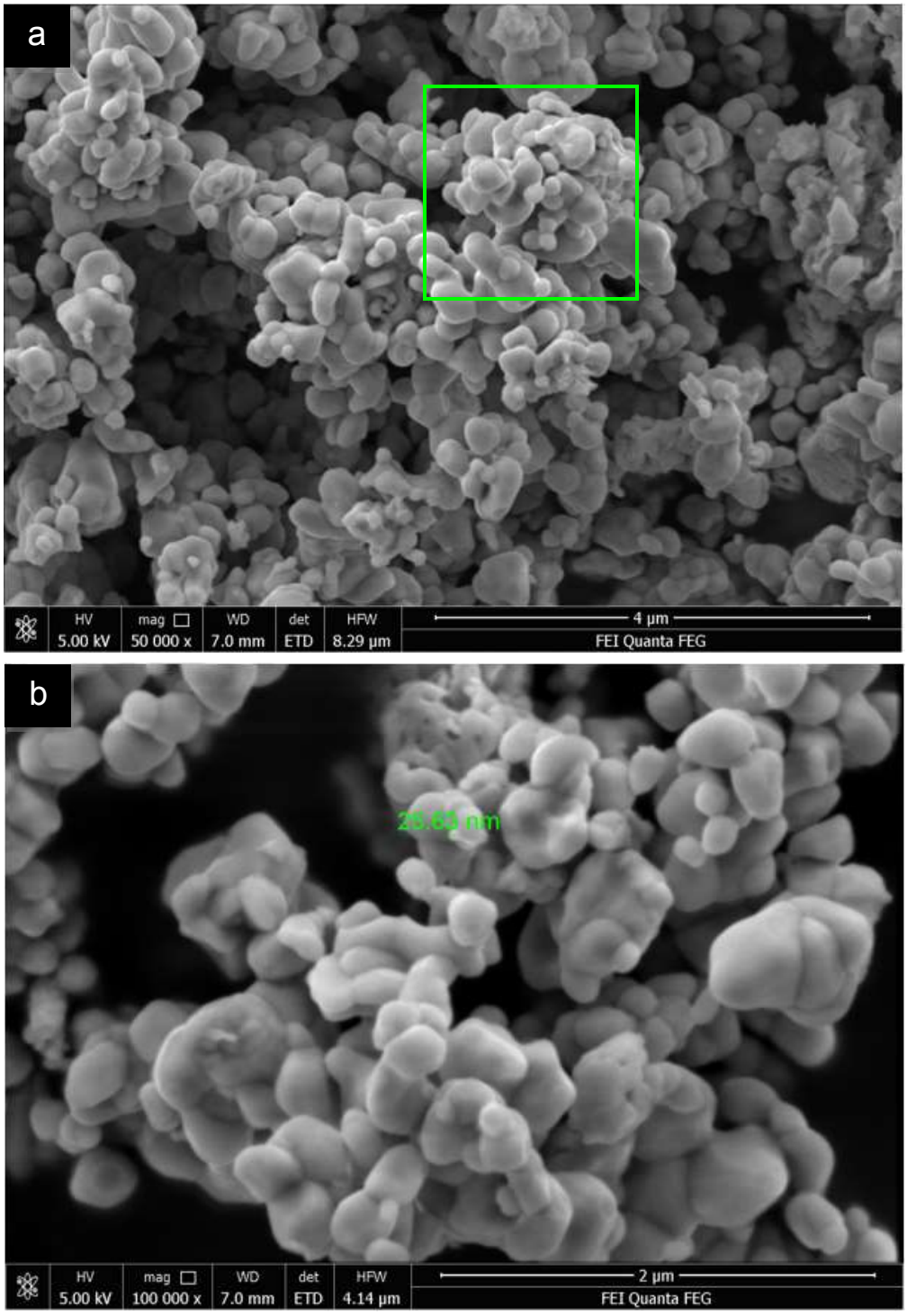
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mag
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WD
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det
ETD
HFW
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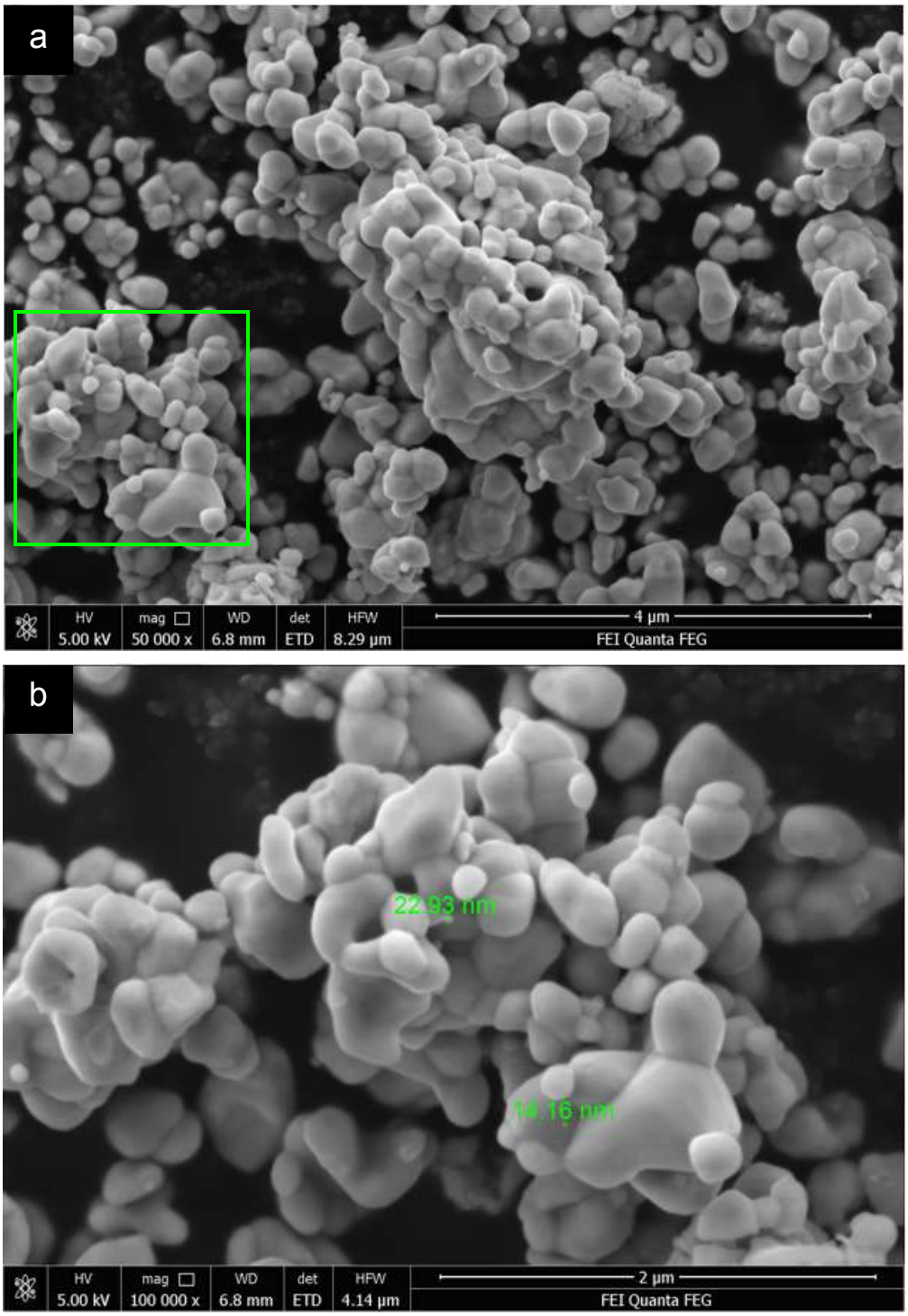
4 µm
FEI Quanta FEG
b
29.63 nm
HV
5.00 kV
mag
100 000 x
WD
7.0 mm
det
ETD
HFW
4.14 µm
2 µm
FEI Quanta FEG

Fig. 9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14.16 nm
HV
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mag
100 000 x
WD
6.8 mm
det
ETD
HFW
4.14 µm
2 µm
FEI Quanta FEG

Fig. 10

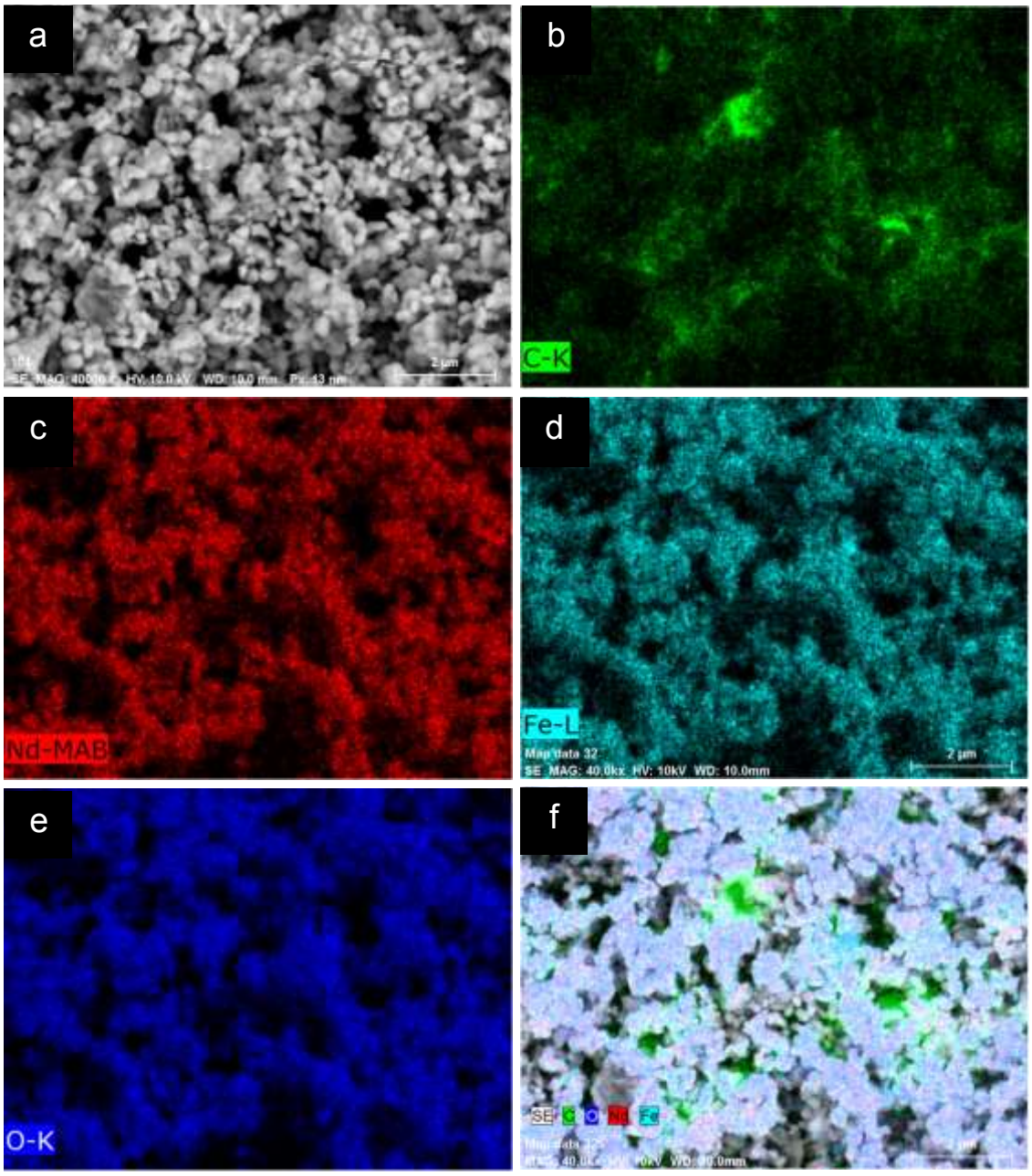
a
b
C-K
c
Nd-MAB
d
Fe-L
Map data 32
SE MAG: 40.0kx HV: 10kV WD: 10.0mm
2 µm
e
O-K
f

Fig. 11

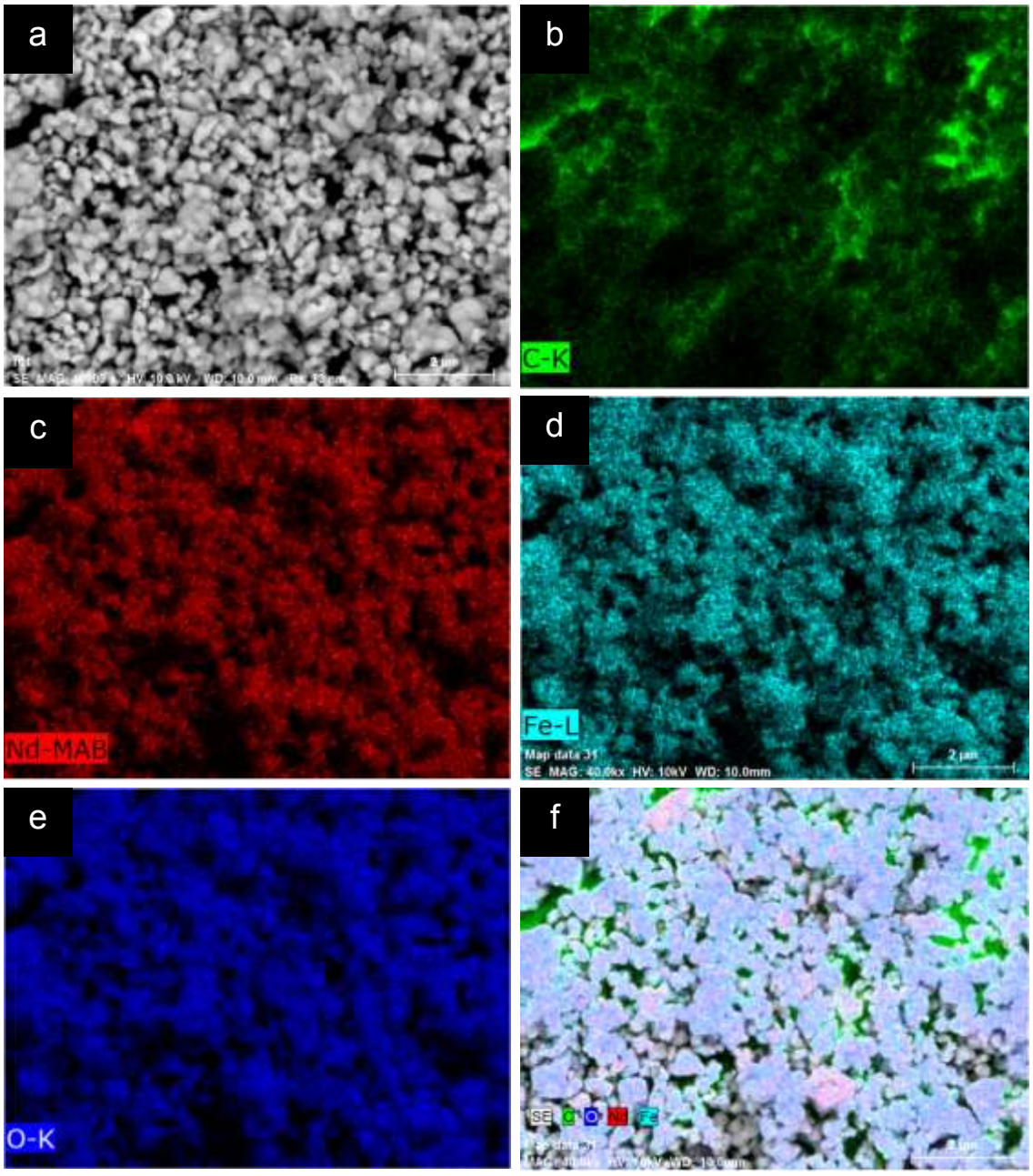
a
b
C-K
c
Nd-MAB
d
Fe-L
Map data 31
SE MAG: 40.0kx HV: 10kV WD: 10.0mm
2 μm
e
O-K
f
SE C O Nd Fe

Fig. 12

Table of Contents

Table 1

Samples	2θ (°)	Intensity (counts)	FWHM (°)	Crystal size (nm)
YK750	32.56	13063.33	0.22 ± 0.5	372.17 ± 0.02
YK850	32.56	12686.67	0.22 ± 0.5	372.22 ± 0.02
YK950	32.56	13050.00	0.22 ± 0.5	372.17 ± 0.02

Table 2

Samples	Profile (Rp)	Weighted Profile (Rwp)	The goodness of Fit (GoF(%))
YK750	4.57	6.06	0.9101
YK850	4.49	6.06	0.8726
YK950	4.73	6.15	0.9303

Table 3

Samples	a (Å)	b (Å)	c (Å)	V (Å^3)
YK750	5.581059 ± 0.000736	7.758627 ± 0.000947	5.448341 ± 0.000665	235.92
YK850	5.580203 ± 0.000695	7.756789 ± 0.000908	5.447646 ± 0.000626	235.79
YK950	5.580402 ± 0.000704	7.758957 ± 0.000919	5.449350 ± 0.000634	235.94

Table 4

Samples	Weight Percentage (%)			
	$NdFeO_3$ Phase	Error	Nd_2O_3 Phase	Error
YK750	99.07	1	0.93	0.1
YK850	99.18	0.96	0.82	0.96
YK950	99.46	0.96	0. 54	0.0

Table 5

Element	Norm. C [wt%]	
	YK 750	YK950
Nd	53.90	49.81
Fe	26.49	23.23
O	15.09	21.35
C	4.52	12.92

Gmail samnur yusuf <samnur74@gmail.com>

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4 pesan

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Kepada: samnur yusuf <samnur74@gmail.com>

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Jl. Daeng Tata Raya Parangtambung
Makassar 90224

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2. table 1 awalnya muncul cuma dua baris data YK 750 & YK 850, jadi saya munculkan yang YK 950
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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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Samnur, ST., MT. (0002057401)

UNIVERSITAS NEGERI MAKASSAR

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Nomor: 1771/UN36.9/PL/2017

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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,sentering 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 Difraction) 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 ditemukenali paramater proses terbaik untuk penumbuhan paduan $NdFeO_3$, bubuk paduan $NdFeO_3$ dan hasilnya antara lain: (1) dua artikel yang telah dipresentasikan pada 2 (dua) forum konferensi internasional yakni International Conference on Applied Material Science anf 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 $NdFeO_3$ dengan doping Pd (Palladium) sebesar maksimum 30 wt%. Proses ini dilakukan untuk menambah sensitivitas $NdFeO_3$ 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 reviuw*) untuk penerbitan ISBN dengan judul Karakteristik Bahan Oksida.

PRAKATA

Segala puji dan syukur atas kehadirat Allah *Azza Wa Jalla* , Rabb semesta alam atas segala rahmat dan nikmatnya yang tidak henti-hentinya dilimpahkan kepada penulis sehingga pembuatan Laporan Kemajuan Penelitian yang berjudul "**Pengembangan senyawa oksida $NdFeO_3$ dan aplikasinya dalam pembuatan sensor gas**" ini dapat diselesaikan.

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 dam 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

DAFTAR ISI

Hal