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Synthesis of ytterbium-doped neodymium ferrite oxide using solid-state reaction method and its characterization

Eko Hadi Sujiono ^{*}, Vicran Zharvan, Muthmainnah Muchtar, Sultra Ade Poetra, Abdi Manab Idris, Muhammad Yusriadi Dahlan, Samnur

Laboratory of Materials Physics, Department of Physics, Universitas Negeri Makassar, Makassar 90224, Indonesia

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ABSTRACT

The Yb doped NdFeO_3 using a solid-state reaction method has successfully synthesized. In this paper, $\text{Nd}_{1-x}\text{Yb}_x\text{FeO}_3$ samples were synthesized by varying the molar ratio of Yb at $x = 0.01$, $x = 0.05$, and $x = 0.10$ using solid-state reaction with two routes of heat treatment processes. Results of X-ray diffraction show that all samples have an orthorhombic structure with two phases: NdFeO_3 as a major phase and Nd_2O_3 as a minor phase. FWHM value for a maximum peak is 0.2° , so the estimated crystal size is 40 nm, with the dominant peak corresponding to hkl (1 2 1). Morphology properties used SEM Image shows grain size of al sample estimated at $0.4 \mu\text{m}$. The presence of Yb is quantitatively confirmed based on the EDS result.

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1. Introduction

ReFeO_3 elements that (Re: rare-earth elements) are known as the rare-earth orthoferrites having orthorhombic structure derived from a perovskite structure [1], and they have attracted much interest due to their properties such as magnetic and magneto-optic [2]. Among these ReFeO_3 elements, NdFeO_3 material useful as a raw material of many applications such as gas sensors, fuel cells, and catalyst material gas sensors [3–6]. The preparation of NdFeO_3 material can realize by several methods [7,8]. The solid-state reaction method has used because this method is cheap and easy to implement. The high purity and crystallinity materials also can be achieved by this method [9].

Present-day, many researchers are working on ReFeO_3 to obtain ideal materials for Adsorbent [10], photocatalytic Material [11], solid-state devices, and gas sensors application [12]. RFeO_3 has a characteristic feature of the presence of two magnetic subsystems of Re^{3+} and Fe^{3+} . The interactions of Fe–Fe, Re–Fe, and Re–Re lead to a few interesting phenomena. ReFeO_3 has the special characteristic of spin reorientations $\Gamma_4(G_x, F_z) \rightarrow \Gamma_{24}(G_x, F_x) \rightarrow \Gamma_2(G_x, F_x)$ [13]. ReFeO_3 shows interesting gas sensitivity properties, gas sensors based on LaFeO_3 show good results to detect ethanol, acetone,

H_2S , CO , and NO_2 [14,15]. In the few past years, NdFeO_3 material as a gas sensor to detect $\text{C}_2\text{H}_5\text{OH}$ [16]. Gas sensors based on ReFeO_3 are developed to detect acetone [17]. The properties of ReFeO_3 elements for gas sensors such as their response and selectivity increased by doping to another oxide material [18] but less information using Ytterbium (Yb) as a dopant element for NdFeO_3 .

In this paper, presented produced the Ytterbium (Yb_x) doped $\text{Nd}_{1-x}\text{FeO}_3$ samples by using a solid-state reaction method by varying the molar ratio of Yb_x from $x = 0.01$, $x = 0.05$ and $x = 0.10$, respectively. All samples then characterized to obtain their crystal structure with qualitative analysis, morphology, and elemental composition properties.

2. Experimental method

The Yb doped NdFeO_3 sample was synthesized using the best parameters found by previous research [18–23]. Raw materials of Nd_2O_3 (Strem Chemicals, 99.99%), Yb_2O_3 (Sigma-Aldrich, 99.99%) and Fe_2O_3 (Sigma-Aldrich, 99.99%) were mixed according to stoichiometric calculation and grinded using mortar for 3 h. That mixed material then calcined using the furnace at temperature 700°C for 4 h. After that, the temperature increased to 950 °C by keeping the temperature constant for 2 h. The heating then decreased to 475 °C and kept it continued for 2 h. After this thermal process, the material was grinded for 6 h.

* Corresponding author.

E-mail address: e.h.sujiono@unm.ac.id (E. Hadi Sujiono).

Material that was obtained ⁹ then calcined at 950 °C for 4 h. After that, the temperature was increased to 1050 °C for 2 h then decreased it to 525 °C for 2 h. All of the processes repeated by varying the Yb content at $x = 0.01$, $x = 0.05$, and $x = 0.10$. The $\text{Yb}_x\text{-Nd}_{1-x}\text{FeO}_3$ samples then characterized using XRD (Rigaku Mini Flex II CuK α , $\lambda = 0.154$ nm) to obtain the phase of crystallographic and SEM-EDAX (FEI Inspect S50) to analyze the morphology and elemental composition, respectively.

7. Results and discussion

3.1. Analysis of x-ray diffraction

Characterization results of x-ray diffraction for Yb doped NdFeO_3 samples can show in Fig. 1. It shows that the pattern has narrow peaks indicating that the samples are in crystalline form. Further analysis using X'Pert High Score Plus software shows the presence of NdFeO_3 and Nd_2O_3 phases. Those phases give information that samples are polycrystalline material [9] with the highest peak lead to the NdFeO_3 phase at the plane (1 2 1) in orthorhombic structure, which is similar to other results [9,16,24]. The presence of Nd_2O_3 phase due to the low temperature during the calcination process [9]. There is no additional peak of impurity observed, which indicates that the samples consist of pure phases [25].

Further analysis of Yb doped NdFeO_3 describes in Table 1. It shows that the 2 θ for peak (1 2 1) gradually shift into a lower degree, comparing to undoped NdFeO_3 [26]. This phenomenon can occur due to lattice distortion [24,27], further analysis of FWHM, 2 θ , and crystalline size shown in Table 1.

It can be seen that increasing of Yb content gradually shift the 2 θ degree into the lower degree and decrease the value of crystalline size from 67 nm as reference value to 40 nm due to the diffusion of ion Yb^{3+} with ionic radii (0.98 Å) less than ionic radii of Nd^{3+} (1.11 Å) as donor into the lattice of NdFeO_3 as acceptor [28].

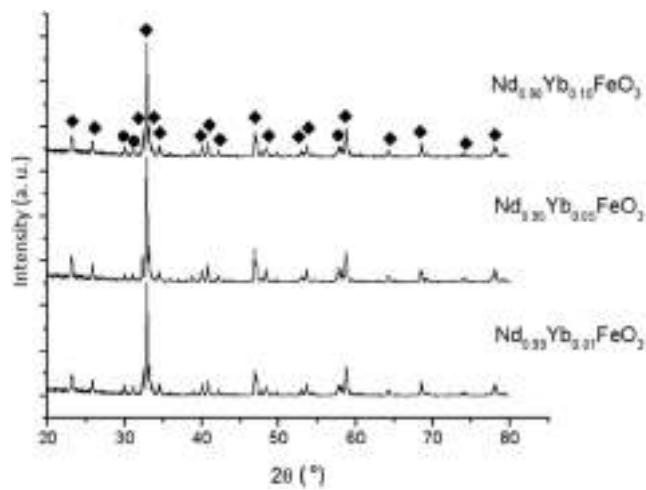


Fig. 1. X-ray diffraction pattern of Yb doped NdFeO_3 (◆ = NdFeO_3 , ● = Nd_2O_3).

Table 1

X-ray analysis results of Yb_x doped $\text{Nd}_{1-x}\text{FeO}_3$ at plane (1 2 1).

X	2 θ (degree)	FWHM (degree)	Crystalline Size* (nm)
0.01	32.8294	0.20197	40.56 ± 0.02
0.05	32.8081	0.20279	40.40 ± 0.02
0.10	32.7940	0.20138	40.68 ± 0.02
0**	32.8600	0.20000	67.00

* Calculated by Debye-Scherrer method**) Khorasani, et al. [26].

3.2. Analysis of SEM-EDAX

The influence of the Yb dopant on the morphology of NdFeO_3 shown in Fig. 2.

The images show all of the Yb-NdFeO₃ powders are quite uniform in shape and size. It gives information that increasing Yb does not provide a significant effect on morphology. The agglomeration also exists for all powders due to the factor of temperature and mechanical treatment during processes [18]. The particle size also can be estimated an average at 0.4 μm from the images. The presence of Yb_x in the $\text{Nd}_{1-x}\text{FeO}_3$ sample can be confirmed based on the results of the EDS spectrum. It can show in Fig. 3.

Based on Fig. 3 shows the presence of Yb ions in the NdFeO_3 sample. The maximum peak of the NdLa spectrum decreases with the increasing YbLa spectrum so that the doping process of the Yb^{3+} ion to the $\text{Nd}_{1-x}\text{FeO}_3$ is successful. Table 2 shows the EDS results for each sample to convince the presence of Yb on the sample and their composition. These results confirmed that the increasing ratio molar of Ytterbium-doped to NdFeO_3 related to Yb amount increase and Nd decreases in elements (wt%), respectively.

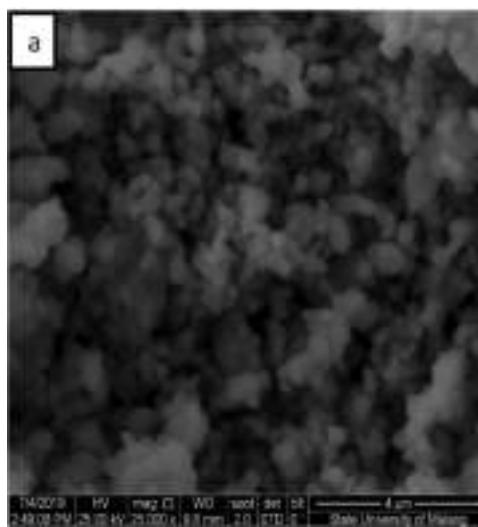


Fig. 2a. Morphology of sample Ybx doped $\text{Nd}_{1-x}\text{FeO}_3$ (a) $x = 0.01$.

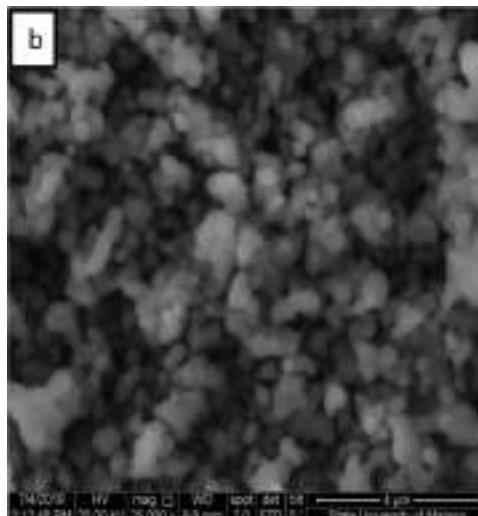


Fig. 2b. Morphology of sample Ybx doped $\text{Nd}_{1-x}\text{FeO}_3$ (b) $x = 0.05$.

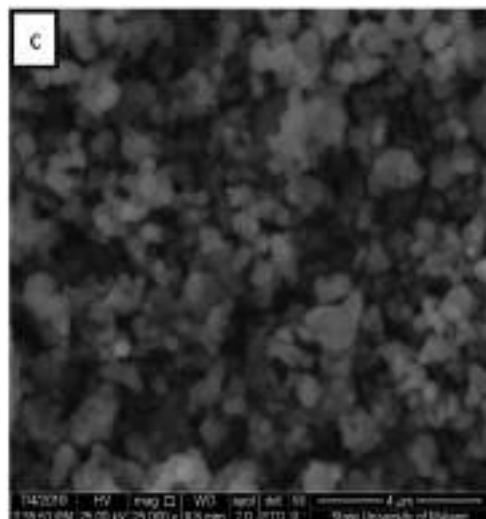


Fig 2c. Morphology of sample Ybx doped Nd_{1-x}FeO₃ (c) $\times = 0.10$.

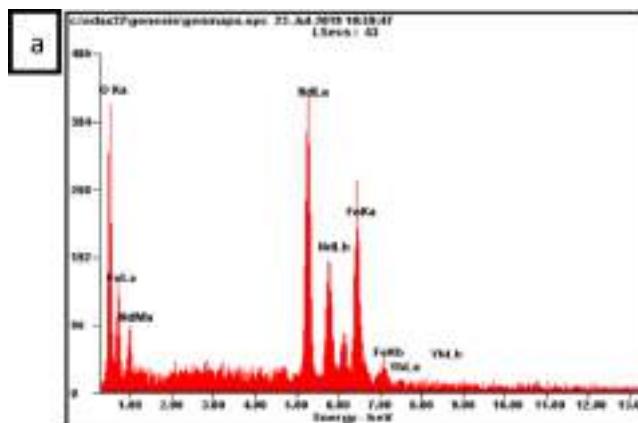


Fig 3a. EDS spectrum of sample Ybx doped Nd_{1-x}FeO₃ (a) $\times = 0.01$.

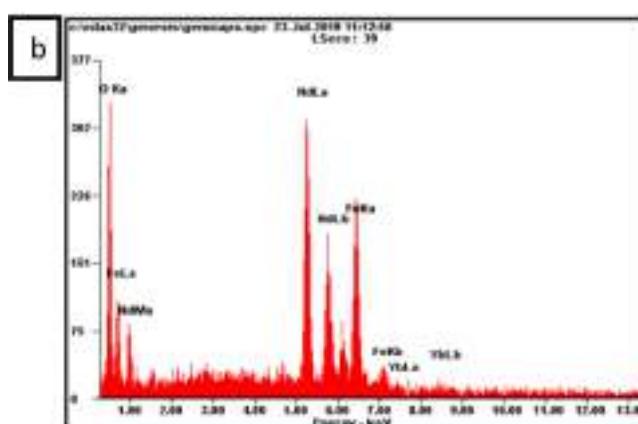


Fig 3b. EDS spectrum of sample Ybx doped Nd_{1-x}FeO₃ (b) $\times = 0.05$.

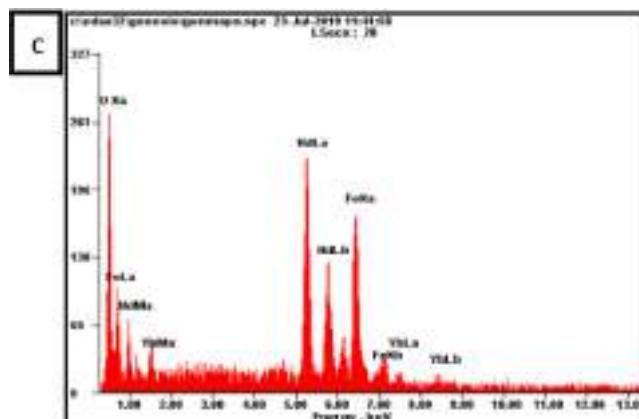


Fig 3c. EDS spectrum of sample Ybx doped Nd_{1-x}FeO₃ (c) $\times = 0.10$.

Table 2
EDS results of the presence of Yb doped NdFeO₃.

X	Elements (wt%)			
	O	Yb	Nd	Fe
0.01	16.66	1.84	60.45	21.05
0.05	16.40	3.15	58.64	21.82
0.10	17.78	8.52	52.18	21.52

4. Conclusion

The Yb-doped NdFeO₃ samples successfully synthesized by using a solid-state reaction method. Data from XRD analysis show that the presence of Yb shifted the 2θ to a lower degree. It can explain because the Yb³⁺ ion, which has less ionic radii than Nd³⁺ ion, successfully substitutes into NdFeO₃ resulting in the distortion on the NdFeO₃ lattice but do not gives a significant effect on their morphology as SEM images confirmed. The grain size of the Yb-NdFeO₃ estimated at 0.4 μm. EDS results show the presence of Yb ions in the NdFeO₃ sample.

CRediT authorship contribution statement

Eko Hadi Sujiono: Conceptualization, Formal analysis, Funding acquisition, Methodology, Resources, Software, Supervision, Validation, Visualization, Writing - original draft, Writing - review & editing. **Vicran Zharvan:** Data curation, Formal analysis, Investigation, Methodology, Supervision, Validation, Visualization, Writing - original draft. **Muthmainnah Muchtar:** Data curation, Formal analysis, Investigation, Methodology, Visualization. **Sultra Ade Poetra:** Data curation, Formal analysis, Investigation, Methodology, Visualization. **Abdi Manab Idris:** Data curation, Formal analysis, Investigation, Methodology, Visualization, Writing - original draft. **Muhammad Yusriadi Dahlan:** Data curation, Formal analysis, Investigation, Methodology, Visualization. **Sammur:** Funding acquisition, Project administration, Resources, Software, Supervision, Validation, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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