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

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ABSTRACT

The Yb doped NdFeO₃ using a solid-state reaction method has successfully synthesized. In this paper, Nd_{1-x}Yb_xFeO₃ samples were synthesized by varying the molar ratio of Yb at \times = 0.01, x = 0.05, and \times = 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: NdFeO₃ as a major phase and Nd₂O₃ 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 µm. The presence of Yb is quantitatively confirmed based on the EDS result.

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1. Introduction

ReFeO₃ 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 magnetooptic [2]. Among these ReFeO₃ elements, NdFeO₃ 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 NdFeO₃ material can realize by several methods [7,8]. The solidstate 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 ReFeO₃ to obtain ideal materials for Adsorbent [10], photocatalytic Material [11], solid-state devices, and gas sensors application [12]. RFeO₃ has a characteristic feature of the presence of two magnetic subsystems of Re³⁺ and Fe³⁺. The interactions of Fe-Fe, Re-Fe, and Re-Re lead to a few interesting phenomena. ReFeO₃ has the special characteristic of spin reorientations $\Gamma_4(G_{xx},F_z) \rightarrow \Gamma_{24}(G_{xz},F_{xz}) \rightarrow \Gamma_2(G_z,F_x)$ [13]. ReFeO₃ shows interesting gas sensitivity properties, gas sensors based on LaFeO₃ show good results to detect ethanol, acetone, H_2S , CO, and NO₂ [14,15]. In the few past years, NdFeO₃ material as a gas sensor to detect C_2H_5OH [16]. Gas sensors based on ReFeO₃ are developed to detect acetone [17]. The properties of ReFeO₃ 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 NdFeO₃.

In this paper, presented produced the Ytterbium (Yb_x) doped Nd_{1-x}FeO₃ samples by using a solid-state reaction method by varying the molar ratio of Yb_x from $\times = 0.01$, x = 0.05 and $\times = 0.10$, respectively. All samples then characterized to obtain their crystal structure with qualitative analysis, morphology, and elemental composition properties.

2. Experimental method

The Yb doped NdFeO₃ sample was synthesized using the best parameters found by previous research [18–23]. Raw materials of Nd₂O₃ (Strem Chemicals, 99.99%), Yb₂O₃ (Sigma-Aldrich, 99.99%) and Fe₂O₃ (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 700C 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.

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Material that was obtained 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 \times = 0.01, x = 0.05, and \times = 0.10. The Yb_x-Nd_{1-x}FeO₃ samples then characterized using XRD (Rigaku Mini Flex II CuK α , λ = 0.154 nm) to obtain the phase of crystallographic and SEM-EDAX (FEI Inspect S50) to analyze the morphology and elemental composition, respectively.

3. Results and discussion

3.1. Analysis of x-ray diffraction

Characterization results of x-ray diffraction for Yb doped NdFeO₃ 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 NdFeO₃ and Nd₂O₃ phases. Those phases give information that samples are polycrystalline material [9] with the highest peak lead to the NdFeO₃ phase at the plane (1 2 1) in orthorhombic structure, which is similar to other results [9,16,24]. The presence of Nd₂O₃ 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 NdFeO₃ describes in Table 1. It shows that the 2θ for peak (1 2 1) gradually shift into a lower degree, comparing to undoped NdFeO₃ [26]. This phenomenon can occur due to lattice distortion [24,27], further analysis of FWHM, 2θ , and crystalline size shown in Table 1.

It can be seen that increasing of Yb content gradually shift the 2θ 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 Yb³⁺ with ionic radii (0.98Å) less than ionic radii of Nd³⁺ (1.11Å) as donor into the lattice of NdFeO₃ as acceptor [28].



Fig 1. X-ray diffraction pattern of Yb doped NdFeO₃ (\blacklozenge = NdFeO₃, \blacklozenge = Nd₂O₃).

Table 1X-ray analysis results of Yb_x doped $Nd_{1-x}FeO_3$ at plane (1 2 1).

Х	20 (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 μ m from the images. The presence of Yb_x in the Nd_{1-x}FeO₃ 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 NdFeO₃ sample. The maximum peak of the NdLa spectrum decreases with the increasing YbLa spectrum so that the doping process of the Yb³⁺ ion to the Nd_{1-x}FeO₃ 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 NdFeO₃ related to Yb amount increase and Nd decreases in elements (wt%), respectively.



Fig 2a. Morphology of sample Ybx doped Nd1-xFeO3 (a) \times = 0.01.



Fig 2b. Morphology of sample Ybx doped Nd1-xFeO3 (b) \times = 0.05.



Fig 2c. Morphology of sample Ybx doped Nd1-xFeO3 (c) \times = 0.10.



Fig 3a. EDS spectrum of sample Ybx doped Nd1-xFeO3 (a) \times = 0.01.



Fig 3b. EDS spectrum of sample Ybx doped Nd1-xFeO3 (b) \times = 0.05.



Fig 3c. EDS spectrum of sample Ybx doped Nd1-xFeO3 (c) \times = 0.10.

EDS results of the presence of Vb doped NdEeO	
ED3 results of the presence of 10 doped NureO ₃ .	

Х	Elements (w	vt%)		
	0	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 2 θ 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 μ m. 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, Formal analysis, Investigation, Methodology, Visualization, Writing - original draft. **Muhammad Yusriadi Dahlan:** Data curation, Formal analysis, Investigation, Methodology, Visualization, Samnur: 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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