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Morphological and Structural Analysis of $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ Oxide Material

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Keywords: Solid-state reaction, Rietveld refinement, heat treatment, $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ system.

Abstract. $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ oxide material have been successfully synthesized by solid-state reaction with modified heat treatment process to improve the heat time efficiency, calcination at 950˚C for six hours, sintering at 975˚C fo six hours, and annealing at 450˚C for six hours, respectively. The X-ray diffraction pattern shows that a single-phase form of $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ is an orthorhombic (Pmmm) structure. The Rietveld refinement analysis found, the lattice parameter are a = 3.8758Å, b = 3.9101Å, and c = 11.7190Å with χ^2 = 1.394%. The SEM-EDAX image shows that the samples are form clusters with size estimates of 10 - 40μm, and the elemental composition of the oxide materials is Ba rich.

Introduction

Oxide-alloy material is one of the interest research topics in material physics and engineering. This material can be applying in daily life since the properties such as the optic, catalytic, electric, and magnetic. Such as Fe_3O_4 and some other oxide, the frequency of dielectric constant is on range 10 Hz – 1 MHz, the other is Y_2BaCuO_5 has critical current density Jc = 10^4 A/cm^2. The research of oxide-alloy material has conducted with various systems. One of them is the BaCuO system (Barium Copper Oxide), such as TlBaCuO (TBCO), YBaCuO (YBCO) [1,2], and NdBaCuO (NBCO).

The NdBaCuO system has some excellent properties if compared to the other BaCuO system. Base on DTA shows how easily Nd dissolves in the BaCuO system rather Y because Y requires a higher temperature. NdBaCuO also has a high critical temperature, Tc = 96 K [3].

$NdBa_2Cu_3O_{7-\delta}$ (δ = 0.09) that has an orthorhombic crystal structure with space group Pmmm. Lattice constants from Rietveld refinement method are a = 3.8622Å, b = 3.9180Å, and c = 11.7711Å with cell volume V = 178.12 Å^3[4]. On the other hands [5] was found that $Nd_{1+x}Ba_{2-y}Cu_3O_6$ (x = 0.01 – 0.1 and y = 0.03 – 0.1) has tetragonal crystal structure (space group P4/mmm) with a = 3.9056Å, c = 11.823Å dan V = 180.39 Å^3. $YBa_2Cu_3O_{7-x}$ and $NdBa_2Cu_3O_{7-x}$ have perovskite orthorhombic structure [6]. It is interesting to examine the NdBaCuO crystal structure because both orthorhombic and tetragonal structures have both CuO layers, which can affect high critical temperatures and superconducting properties [7,8].

The influence of dopant additions also contributes to BaCuO's crystal structure, with x additions on $Y(Ba_{1-x}Sr_x)_2Cu_3O_w$, O_4's coordinate (one of oxygen in CuO layer) changed from (0, ½ , 0) to (x, ½, 0), the distance between Ba/Sr – O_4 decreases as Sr content increases [9]. This dopant selection base on dopant's ionic radii smaller than the doped material and both are bivalent. Other than that, Fe_2O_3 choose as a dopant to increase magnetic properties for NdBaCuO. Besides the structure, particle size also one of the keys for oxide-alloy material [10]. The NBCO oxide material with the smaller grain size, higher critical current density is caused by improving surface contact area [11]. Grain size and shape also affect angle change between copper oxide bonds [7].

Several methods have been used to synthesized NdBaCuO, such as TSSG, Polymerized complex, sol-gel, and solid-state reaction method. All of this method took much time for the heat treatment process, such as calcination, sintering, and annealing, with various time and the different

molar ratio [3, 10, 12, 13]. In recent research, perovskite oxide only took 6 – 24 h for the heat treatment process [14,15].

Therefore, information on the morphological and structural of oxide material is crucial to know the particle size. This paper described the characterization results of $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ oxide material using XRD, and SEM-EDAX. The effect of the heat treatment process related to the size and morphology information has discussed.

Experiment

$Nd_1Fe_xBa_{2-x}Cu_3O_{7-\delta}$ has prepared by a solid-state reaction method. High purity of Nd_2O_3 powder (99.99%), $BaCO_3$(99.98%), CuO (99.99%), and Fe_2O_3(99.98%) were mixing and grinding together with molar ratio x = 0.2 in mortar and pestle for 3 hours to reach homogenous powder. The precursor was calcined at 950℃ for 6 hours and regrinding for 5 hours [16]. Then, the mixture was sintered at 975°C and annealed at 450℃, each for 6 hours with oxygen flow [16].

Analysis of the XRD pattern was carried out using Match!3 software to know the phase and crystallography data. Furthermore, the Rietica software used for Rietveld refinement analysis of the lattice constants and Full-Width at Half Maximum (FWHM). After refinement analysis, the data used Diamond software for visualized and described the distance between atoms also the angles.

Result and Discussion

X-Ray Diffraction Analysis

After the heat treatment for 18 h, $Nd_1Fe_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ characterized by XRD. Peak pattern, as shown in figure 1, there are 11 peaks identified by the software. The Match! 3 has used, firstly we search for a similar pattern and the same phase as we have. Since there is no one similar phase database on Match! So we decided to the most similar peak pattern in our sample. It was $NdBa_2Cu_3O_{6.92}$ [12]. The most dominant peak was at 2θ° 32.41° (110/013), with an intensity of 570 cps. Crystallography data shows that the structure is orthorhombic with space group Pmmm.

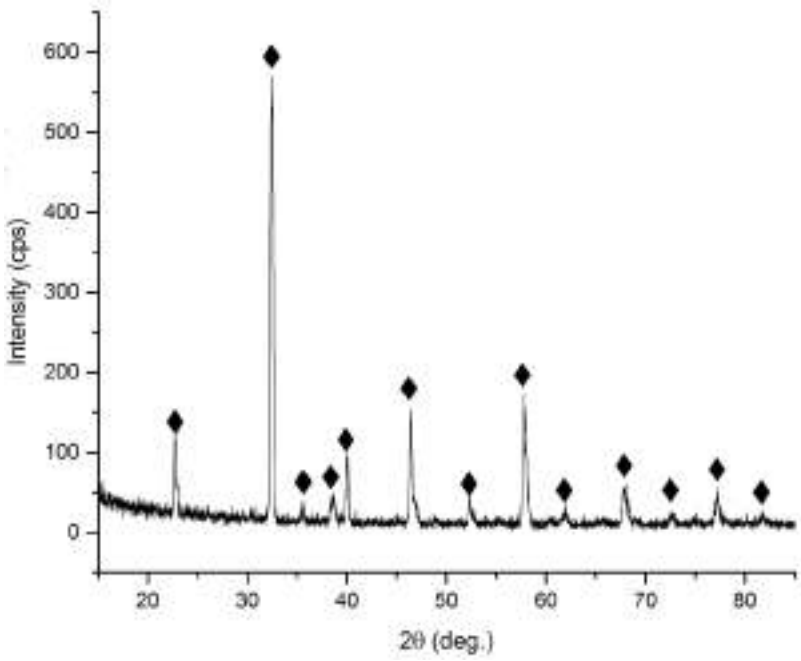

Fig. 1 XRD pattern of Nd(Fe)BaCuO (♦ = Nd(Fe)BaCuO).

To describe the crystal structure, the refinement performed by the Rietveld method using Rietica software. Figure 2 shows the calculated pattern noted with a red curve, similar to the measured pattern illustrated with the black curve. Table 1 presented the value of χ^2 is 1.394, and the χ^2 value of less than 4 is an accepted constant factor of refinement [17]. The atom occupancy of Ba decrease

from 0.250 to 0.247. It caused the addition of Fe dopant as we know Fe ionic radii smaller than Ba, also confirmed that the volume cell smaller than has been reported [12]. The visualization of $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ oxide structure shown in figure 3. The most exciting aspect of the configuration is the existence of angular change between the copper oxide bonds (Cu-O-Cu layer) becomes more pointed when viewed from the a-axis (151.932°) and b-axis (141.962°). This change will affect the superconducting properties [7].

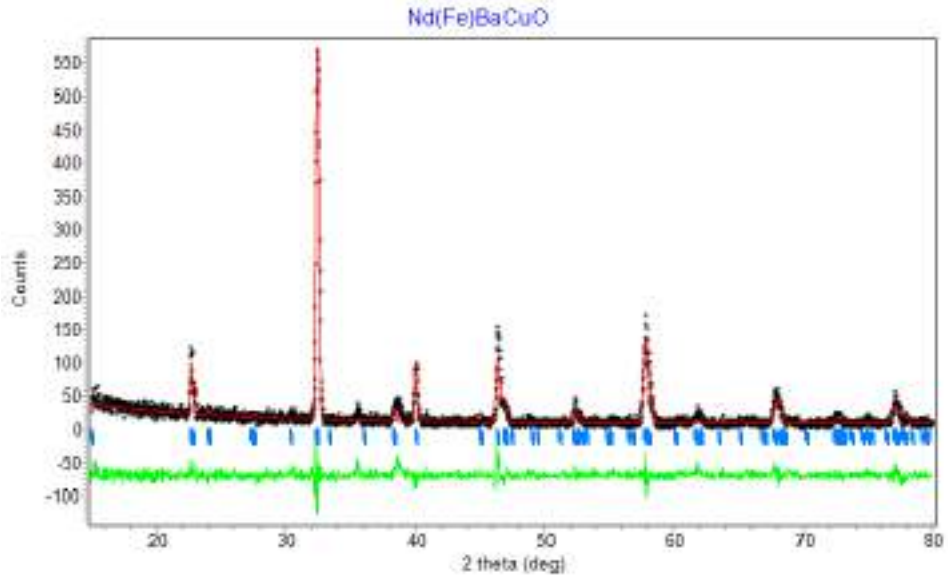

Fig. 2 The plot of the Rietveld refinement results by using Rietica for Nd(Fe)BaCuO at 2θ of 10-80º.

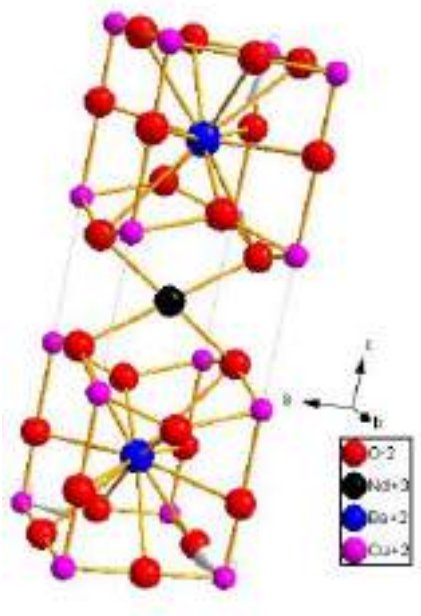

Fig. 3 Visualization atomic structure of Nd(Fe)BaCuO phase with molar ratio x = 0.2 and the lattice parameter is a = 3.8758Å, b = 3.9101Å, c = 11.7190Å.

Rietveld refinement also has a general parameter (U, V, W) that we have refined. Using this parameter, the FWHM defined with the following equation (1):

$$H = \left(U \tan^2 \theta + V \tan \theta - W\right)^{\frac{1}{2}} \quad (1)$$

Which is H indicate of FWHM (Full Width at Half Maximum) the crystalline quality of oxide materials, smaller FWHM means higher crystalline quality [14]. The FWHM of this sample is 0.266°, and the crystal size is D = 4.952 Å.

Table 1 Refinement parameters of Nd(Fe)BaCuO oxide material.

$Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$	
Space group	Pmmm
Lattice constants	
a	
b	3.8758 Å
c	3.9101 Å
	11.7190 Å
Volume	177.5987 $Å^3$
n_{Ba}	0.247
R_{wp}	25.05
R_p	18.74
χ^2	1.394

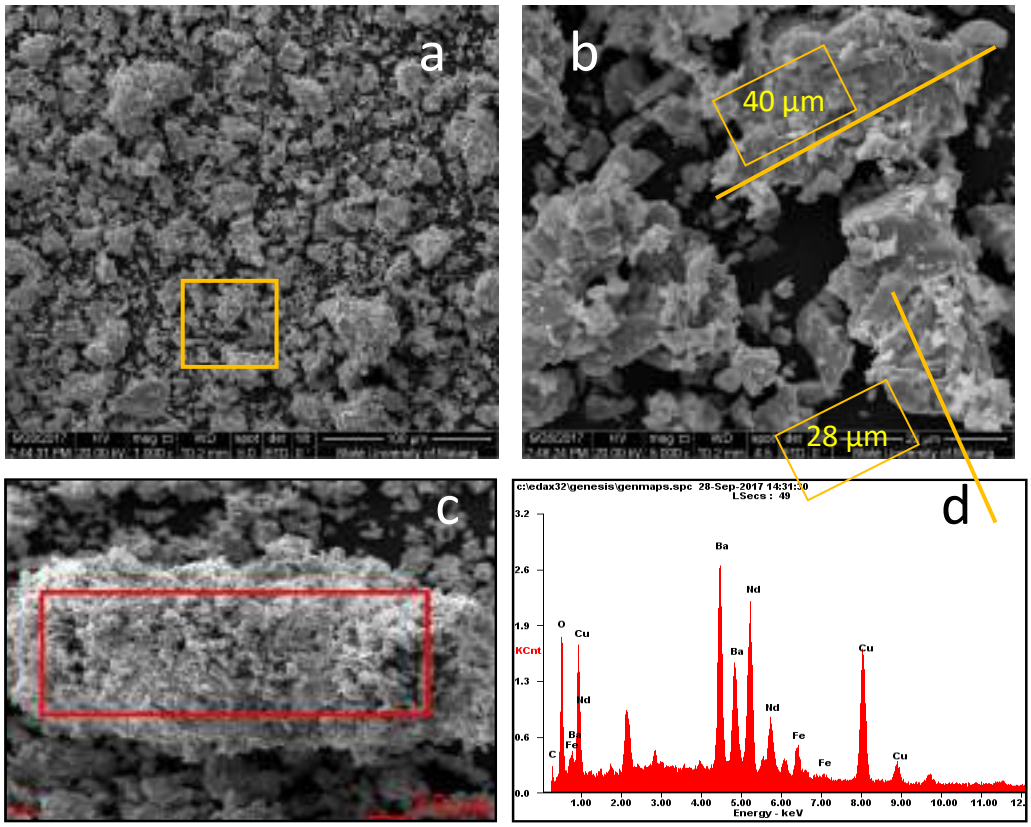

Fig. 4 SEM images of sample Nd(Fe)BaCuO phase with the molar ratio of x = 0.2, a) the magnification is 1,000X, b) the magnification is 5,000X, c) the analyzed area marking with red rectangular, and d) EDAX spectrum of Nd(Fe)BaCuO sample.

SEM-EDAX Analysis

The SEM-EDAX image, as shown in figure 4 Nd(Fe)BaCuO, allegedly formed clusters with estimated size ranging from 10 – 40 µm. The inhomogeneous clusters size probably caused by the heat treatment process of the powder sample. The image in figure 4b presents Nd(Fe)BaCuO grain size with the surface area marked by the heavier atoms that have lighter imaging. It was also marked in figure 4c related to the diffraction pattern of EDAX, as shown in figure 4d. This result confirmed that the sample has Ba-rich.

Summary

The $Nd_1(Fe)_{0.2}Ba_{1.8}Cu_3O_{7-\delta}$ has an orthorhombic structure with space group Pmmm. FWHM counted is 0.266°, and the crystal size is 4.952 Å. The Rietveld refinement method found the lattice parameter a = 3.8758Å, b = 3.9101Å, and c = 11.7190Å with reliable factor χ^2<4. Dopant addition Fe/Ba affected the distance of the atoms, the formation of Cu-O-Cu layer; this phenomenon can affect the superconducting properties. The surface morphology of the sample shows the size of the cluster ranging between 10 – 40 µm with elemental composition is Ba-rich confirmed by EDAX data. The morphological and structural analysis indicates the time efficiency for the heat treatment process has found.

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