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Crystal Structure and Morphology Analysis of $Nd_{1+x}Ba_{2-x}Cu_3O_7$ Oxide Alloy Surface Developed by Solid State Reaction Method

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Abstract

This research has successfully prepared $Nd_{1+x}Ba_{2-x}Cu_3O_7$ oxide alloy which was developed using solid state reaction method with varying molar ratio x = 0.1 to x = 0,3. The process parameter observed was various synthesis routes that were different calcinations temperatures and sintering. In order to acquire the quality of the alloy, the homogeneity of the alloy were analyzed based on the result of EDAX (Energy dispersive X-ray). The composition of the material was analyzed in every variation of synthesis. Last, the crystal and morphology of $Nd_{1+x}Ba_{2-x}Cu-3O-7$ oxide alloy were analyzed based on the result of XRD (x-ray Diffraction) and SEM (Scanning Electron Microscope). The XRD measurements showed that crystal NdBaCuO has been formed. From the results of SEM images also showed that the greater the molar ratio x the particle size will be smaller.

KEYWORDS: solid state reaction, X-Ray diffraction, Scanning Electron Microscope (SEM)

I. INTRODUCTION

Materials science and engineering is the main foundation of modern technological developments, both for the application of structural, electronic, thermal, electrochemical, environmental and biomedical. Each era is characterized by the discovery of new materials. Mechanical properties of materials are mainly influenced by the process of synthesis and composition of materials [1].

The best way to know the quality of a material is to conduct a test or characterization in accordance with the properties that needed to know. For example, a mechanical testing used to investigate the mechanical properties of materials such as compressive strength and Modulus Young. I-V meters are used to investigate the characteristics of strong currents voltage. As for the macroscopic properties of alloy materials such as crystal structures and morphology, an X-ray diffraction (XRD) and scanning electron microscope (SEM) were used.

In this study the oxide alloy materials are particularly to the process of synthesis and analysis of crystal structure and morphology, so the principal problem faced by researchers in the assessment NdBCO oxide alloy materials as superconducting materials such as determining the appropriate synthesis route to produce samples with the best crystal and structure of the surface morphology and the determination of the x value variations in the alloy oxide NdBCO.

$Nd_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$ (NBCO) has a temperature high T_c, when varying Nd and Ba with a low concentration and reduction of critical temperature (T_c) and current density (J_c) depends on Oxygen stoichiometry shown in orthorombik-tetragonal crystal structure on NBCO crystalline phase transition [2].

The aim of this study is to determine the value of x that produce variations in the sample with the best quality, as seen from the homogeneity of the compound, a good crystal structure, and a uniform grain size. By comparing several samples with variations in the value of x that has been in the synthesis and in characterized which produce samples with the best quality.

II. EXPERIMENTAL METHOD

Alloy materials $Nd_{1+x}Ba_{2-x}Cu_3O_7$ with the variation of molar ratio x = 0.1 to x = 0.3 is developed by solid state reaction method. Starting from the mixing of powders of $BaCO_3$ (99.99%), Nd_2O_3 (99.99%), and CuO (99.99%), and then calcinations for 50 hours at a temperature of 950°C. Alloy material is then made in bulk form with a diameter of 1 cm, and height 5 mm, sintering and annealing at a temperature of 950°C and 400°C.

Crystal structure of this alloy was analyzed based on the characterization results of XRD (X-ray diffraction) which includes the value of FWHM (Full Widht at Half Maximum) and peak height. The surface morphology analyzed based on the results in SEM (Scanning Electron Microscope) with a magnification of 1,000 and 10,000 times, including shape, size, and grain growth.

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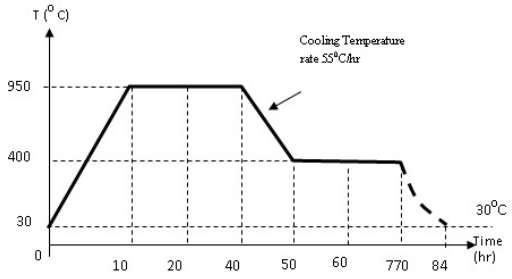

FIG. 1: Sintering and Annealing Graphic

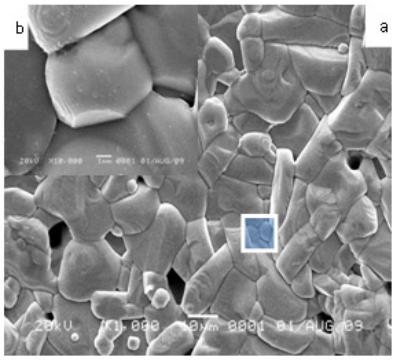

FIG. 2: SEM result for molar fraction x = 0,1 with magnifications (a) 1.000 times, (b) 10.000 times

III. RESULT AND DISCUSSION

A. SEM Measurements

Samples with variation of x = 0.1 (figure 2.b) show almost uniformly square forms in comparison with samples with a variation of 0.2 and 0.3 (Figure 3 and 4). Imperfect grain growth (truncated form) can be seen in the sample with variation x = 0.2 (Figure 3.b). It was caused by uneven heat distribution that resulting heat rate is not constant across the sample surface.

The overall pattern of grain growth for this sample is spiral

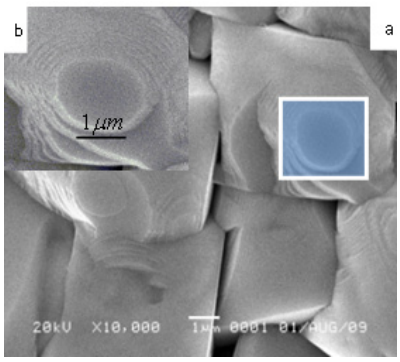

FIG. 3: SEM result for molar fraction x = 0,2 (a) magnifications 1.000 times, (b) truncated form

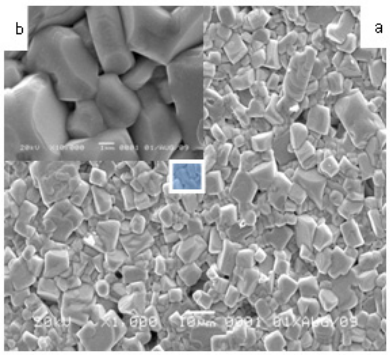

FIG. 4: SEM result for molar fraction x = 0,3 with magnifications (a) 1.000 times, (b) 10.000 times

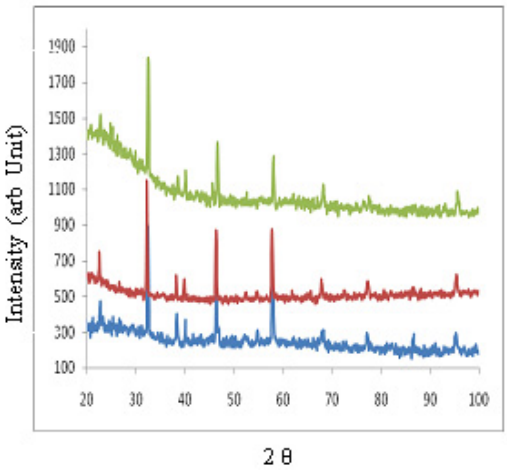

FIG. 5: XRD result for molar fraction x = 0,1 to x = 0,3

growth (as shown in Figure 2.b.). The spiral growth is the twisted form of the grain. Spiral Growth development was previously been observed by Mitzumaza. [3]

B. XRD measurements

Spectrum X-Ray Diffraction (XRD) of the surface alloy materials NdBaCuO to variation of molar ratio x = 0.1 and with x = 0.3 shows the three most dominant peaks for each variation of x (Figure 5).

With the formation of these peaks indicate that the alloy material is synthesized NdBaCuO have shaped the structure of crystals. The difference in height and width of the peak in because of the variation of molar ratio (x) given for each sample. Crystalline quality of a thin film can be seen from the FWHM of the peak area fraction. FWHM stated level of strain on the film, the narrower the FWHM, the smaller strains formed. The most dominant peak for sample 0.1, 0.2, and 0.3 have FWHM of each successively at 0.3, 0.24, 0.28, as shown in table 1.

Table 1 show that there is a variation of the peak and the reflection angle for each variation of the sample. The sample

TABLE I: Value of FWHM for every variation

Molar Ratio (x)	Peak	Intensity	FWHM (2θ)
0,1	320	889	0,3
0,2	320	746	0,24
0,3	320	1033	0,28

with the smallest FWHM value is a variation of x = 0.2, indicating that these samples based on XRD data that have the most orderly arrangement of crystals because the smaller the FWHM value and width peak (Tip Width) the grids are more homogeneous or more orderly arrangement of crystals. This means that the level of material quality is also better. Wickenden, states that a thin film with FWHM values below 0.50 is good crystalline quality.[4]

XRD characterization results showed that the most dominant peak for all the variations of x is at an angle 2θ = 32.4502° with the intensity of 1033 cps (fig.5). The higher the intensity of the XRD measurements showed that the better crystallization material. Based on this, we can say that this sample also has the best conductivity or the least resistivity [5], it is also strengthened by the results of SEM that shows the sample with x = 0.3 has the smallest particle size (figure 4.a) Based on research conducted by Wisnu Ari Adi, et al. In their study entitled "Effect of Powder Size on Critical Current Density Superconductors $NdBa_2Cu_3O_{7-x}$ " in 2003 which concluded that the smaller the size of the powder, the higher the density of the sample, causing greater price J_c [6].

IV. CONCLUSION

The amount of variation given in molar stoichiometric alloy materials $Nd_{1+x}Ba_{2-x}Cu_3O_7$ greatly affect the morphology of the surface of materials such as grain size, the appearance of grain boundary and grain-growing form. From the result of XRD spectrum, this also greatly affects the crystal structure which was indicated by the change in width and height of the peak. When the value of molar ratio increase, the size of grains that are formed will decrease, but for the peak width of XRD spectrum will become bigger along with the increase of molar ratio x.

Acknowledgments

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