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INFLUENCE OF SUBSTITUTION OF GD ON CA ELEMENTS IN PHASE 2212 SUPERCONDUCTOR: $BI_2SR_2(GD_XCA_{1-X})CU_2O_Z$

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ABSTRACT

In this research, the influence of substitution of Gd on Ca on Bismuth superconductor phase 2212 has been successful. The synthesis process is done by using wet-mixing method. The synthesis follows the steps: weighing, mixing with HNO_3, calcination and sintering. Characterization is done with X-ray radiation (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning electron microscope (SEM). The XRD results show a spectrum dominated by high-intensity $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$ occurrences and phase impurities of 2201 ($Bi_2Sr_2CuO_x$) and phase 21 (Ca_2CuO_3) with little intensity. The addition of the Gd element causes a change of the lattice parameter, so that the unit cell volume becomes larger. In contrast, the addition of the Gd element causes the atomic density in the unit cell to become smaller. The SEM characterization results show rod shaped with particles of size from 30 to 101 nm. The FTIR characterization results show the wave absorption at the wave number of 439.77, 632.65, 954.76, 1145.72, 1269.16, 1716.65, 3001.24, 3714.90 and 3844.13 cm^{-1}. These wave numbers are related to bending vibration absorption of CO_3^{2-}, vibration M-O and stretching C-H.

KEYWORDS

superconductor, Bi-2212 phase, wet-mixing

1. Introduction

Applications of high-temperature superconducting materials vary widely, such as electronic device applications, magnetic levitation, superconducting magnetic energy storage systems (SMES), etc., which are produced on a large scale or laboratory scale [L. Rostila 2006, P. Tixador 2010]]. High-temperature superconductors capable of contributing to good applications are the Bismuth (BSCCO) system. The problem is that the critical magnetic field of the material is still too low. In this study, we examine the effect of substitution of Gd on Ca on the formation of $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$ superconductor.

System of superconductor BSCCO that have the general formula $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ are the most anisotropic superconductors among other high-temperature superconductors. The anisotropic state in $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ seems to be caused by a very weak coupling between the BiO-BiO double layers. From the above compound formula, the system of superconductor BSCCO has three phases related to an (1, 2, 3) namely: phase 2201, phase 2212 and phase 2223. In a system of superconductor BSCCO, it is known that the superconductivity phenomenon occurs in the layer / field of Cu-O, Sr-O is an insulator, while the Bi-O layer is semiconductive.

Giving doping (replacement) and flux greatly affect the properties of superconductor. For example, doping in the case of superconductor $Bi_2Sr_2CaCu_2O_y$ as reported by Nguyen Thuy Sinh and friends [Nguyen 1995], Ca element can be substituted by element Y. In a certain limit at x = 0.2-0.35, its Tc price becomes maximum. Then decreases with increasing price x. This indicates a dependence on doping hole where the dependence is close to parabolic. Doping / flux of Pb on Bi superconductor on a phase 2223 system is also very effective for removing impurities so as to produce single crystals. Nd doping of the Y123 superconductor system has also been shown to increase critical temperature [N. Mori 2006]. Then the system was developed again in 1212 phase which was first discovered by A. Ehman and his friends [A. Ehman 1993] with composition $(Bi,Cu)Sr_2YCu_2O_x$ with critical temperature Tc ~ 68 K.

The weakness of the superconductor is that it has a magnetic field price is very small, so it will have a bad impact on the application. Therefore, in this research will be substituted of Gd elements in Ca, in hopes to increase the magnetic field value of superconductor Bismuth on phase 2212, so that in its application do not require high cost. The Gd element is used for substitution of the superconductor Bismuth system, because the Gd element is a rare earth element which is a magnetic material.
This experiment was designed to examine the problem of how the substitution effect of Gd with Ca on the formation of Bismuth-based superconductor with phase 2212. With the substitution is expected to change the mechanical properties of materials: weak link, lattice strain and doping hole so that there is a change to the structure of phase crystals 2212, and its conductivity behavior. With these changes it is expected that the material becomes superconductor with high Tc and is able to increase the critical magnetic field of the material. By observing these behavioral changes can be studied what and how the role of each of the elements in the material in the mechanism of superconducting occurs.

So the specific purpose of this study is to know how the effect of substitution of Gd elements on the micro structure (lattice parameter, cell volume and reliability factor) and homogeneity of superconductor $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$.

The significance of this research is the substitution of Gd elements in the superconductor Bismuth system phase 2212 ($Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$), which will be observed changes in the microstructure of the material, such as changes in lattice parameters, volume fraction and reliability factors based on the results of XRD refinements. While the homogeneity of the sample in terms of particle size was observed based on the calculation result using Scherrer equation.

In this research, the synthesis process is done by the wet mixing method. The wet mixing method has been done by many researchers, such as to cultivate YBCO superconductor crystal with the substitution of rare earth elements [WG Suharta 2013] and growth of NLBCO with the substitution of B_2O_3 flux [WG Suharta 2016].

2. Experiment

The starting materials, which are chemicals in powder form, are Bi_2O_3, $SrCO_3$, $CaCO_3$, CuO and Gd_2O_3 with a purity of 99.9% to avoid contamination of other elements. The synthesis process used in this research is by using wet chemical method, with the following steps:

- The mixture is made in a molar ratio, then the molar composition is made in the form of a heavy percentage.
- The mixture was dissolved with HNO_3 and stirred using a magnetic stirrer and heated at 50 ° C for 20 minutes.
- The three solutions are mixed and stirred using a magnetic stirrer and heated to a temperature below 100 ° C to the crust.
- The compound in the crust form is cured at 100 ° C for 1 hour to remove carbonic acid (H_2CO_3).
- The compound of the oven is calcined at 600 ° C for 3 hours to separate the nitrate content.
- Sintering process is carried out at 850 ° C with sintering time variation from 2 to 10 hours to eliminate the phase of impurities.
- Characterization of samples that have been successfully created,

tested and characterized by X-ray Diffraction (XRD), scanning electron microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR).

- Analysis of diffraction data is done by matching and refinement of the diffraction pattern.

3. Result and Discussion

$Bi_2Sr_2(Gd_{1-x}Ca_{1+x})Cu_2O_z$ samples that have been successfully synthesized using the wet-mixing method is characterized by X-ray diffraction (XRD) to see the success of the established phases. Result of XRD characterization for sample $Bi_2Sr_2(Gd_{0.05}Ca_{0.95})Cu_2O_z$, $Bi_2Sr_2(Gd_{0.10}Ca_{0.90})Cu_2O_z$, $Bi_2Sr_2(Gd_{0.15}Ca_{0.85})Cu_2O_z$, $Bi_2Sr_2(Gd_{0.20}Ca_{0.80})Cu_2O_z$ and $Bi_2Sr_2(Gd_{0.25}Ca_{0.75})Cu_2O_z$ is shown in Figure 1. The spectrum shows a high-intensity and sharp peak of $Bi_2Sr_2(Gd_{1-x}Ca_{1+x})Cu_2O_z$ which indicates good crystallization.

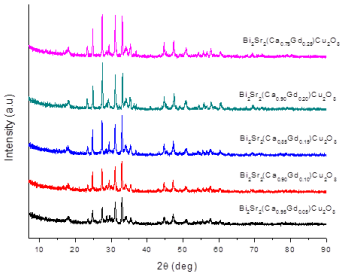

Figure 1. Result of characterization XRD sample $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$

The results of the synthesized characterization of XRD samples were analyzed using the Match program to ensure the phase formed in the sample. The Match program is a software capable of analyzing the phases formed in a solid. The Match process, known as the diffraction peak matching process, refers to the reference powder diffraction file (PDF) that matches the sample. The results show the presence of $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$ superconductor at each 2θ angle with high intensity, while also showing the presence of 2201 ($Bi_2Sr_2CuO_x$) and 21 (Ca_2CuO_3) phases with low-intensity as impurity phase.

The addition of the Gd element to the $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$ superconductor produces the shift of the X-ray diffraction peak at each angle 2θ. The shift of the X-ray diffraction peak is caused by the difference of the radius of Gd ions with Ca. Angle 2θ The difference in radius microscopically leads to changes in the dimensions of the crystal structure. To be able to see clearly the angular shift in the sample, the XRD characterization results are formed in a short angle. The angular shift between 22-28° is shown in Figure 2, which is the angle shift for the $BS(G_xCa_{1-x})CO$ sample. While the angular shift of 2θ for the samples at angles between 28-35° is shown in Figs 3. The diffraction peak shift occurs from a small angle to a large angle with the addition of substitution of the Gd element.

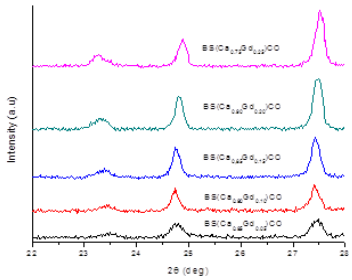

Figure 2. The shift angle of the sample diffraction peak $Bi_2Sr_2(Gd_xCa_{2-x})Cu_2O_z$ between 22-28°

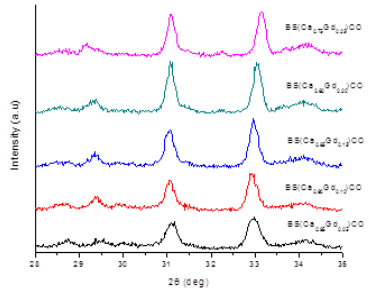

Figure 3. The shift angle of the sample diffraction peak $Bi_2Sr_2(Gd_xCa_{2-x})Cu_2O_z$ between 28-35°

The method of refinement or pattern diffraction matching of the model with the observed diffraction pattern using the least squares method is one of Rietveld's analyzes. The degree of compatibility between the calculated diffraction pattern and the observations is expressed by Rwp (weighted profile factor), Rp (profile factor), S (goodness-of-fit), R_B (Bragg factor).

From the results of refinement of all samples obtained value of lattice constant toward axis-a, b-axis and c-axis. Also obtained the value of reliability, volume and density of atoms in unit cell. These parameter values are shown in Table 1.

Table 1. The value of parameter values (a, b, c), reliability, volume and unit cell density

Sample	Lattice parameters			Reliability					Density (g cm^{-2})
	a (Å)	b (Å)	c (Å)	R_p	R_{wp}	R_e	GoF	Volume ($Å^3$)	
$(BS(C_{1.95}G_{0.05})CO$	5.4109(6)	5.4177(5)	30.5137(6)	21.99	28.37	16.53	2.94	894.498	15.453
$(BS(C_{1.90}G_{0.10})CO$	5.4176(1)	5.4252(9)	30.4756(1)	27.98	27.98	16.43	2.90	895.726	15.437
$(BS(C_{1.85}G_{0.15})CO$	5.4234(0)	5.4381(6)	30.4171(9)	20.52	26.37	16.39	2.61	897.091	15.430
$(BS(C_{1.80}G_{0.20})CO$	5.4253(4)	5.4589(8)	30.3950(2)	19.45	25.09	16.79	2.23	900.183	15.376
$(BS(C_{1.75}G_{0.25})CO$	5.4286(3)	5.4697(5)	30.3923(8)	20.26	26.30	17.14	2.36	902.433	15,360

The result of lattice parameter shows that the addition of Gd element causes the parameter of lattice toward the a-axis and the b-axis are bigger, while the lattice parameter value toward the c-axis decreases, as shown in Table 1 and Figure 4. In addition, the volume of the unit cell increases and the unit cell density decreases with the addition of the Gd element.

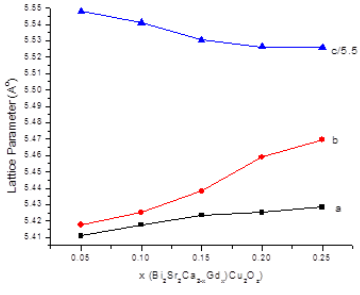

Figure 4. The lattice parameter change curve (a, b, c) $BS(C_{1.75}G_{0.25})CO$

The FTIR characterization results for the $BS(C_{1.95}G_{0.05})CO$, $BS(C_{1.90}G_{0.10})CO$ and $BS(C_{1.85}G_{0.15})CO$ samples are shown in Figure 5. The presence of the absorption peaks present in the sample is in the range of wave numbers 439.77, 632.65, 954.76, 1145.72, 1269.16, 1716.65, 3001.24, 3714.90 and 3844.13 cm^{-1}. These wave numbers are related to bending vibration absorption of CO_3^{2-}, vibration M-O and stretching C-H. The resulting pattern is almost the same and no significant change is obtained.

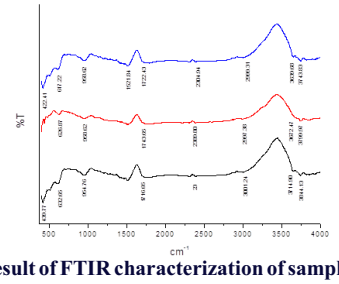

Figure 5. Result of FTIR characterization of sample $Bi_2Sr_2(Gd_xCa_{2-x})Cu_2O_z$

The SEM characterization results for the $Bi_2Sr_2(Gd_{0.05}Ca_{0.95})Cu_2O_z$, and $Bi_2Sr_2(Gd_{0.10}Ca_{0.90})Cu_2O_z$ samples are shown in Figures 6a and 6b. The characterization was performed by enlarging 25,000 times at 20kV energy showing images on a 1 μm scale. In general the shape of a particle is a rod shape (shaped bar).

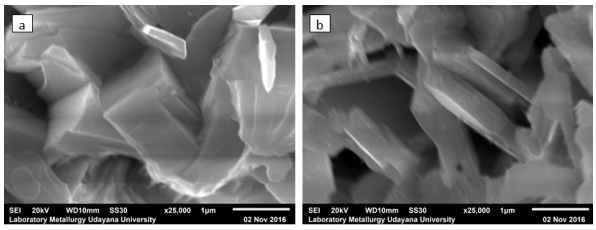

Figure 6. Results of SEM characterization of sample a) $Bi_2Sr_2(Gd_{0.05}Ca_{0.95})Cu_2O_z$ and b) $Bi_2Sr_2(Gd_{0.10}Ca_{0.90})Cu_2O_z$

The result of calculation of particle size by using Scherrer equation and ImageJ programs for $BS(G_{0.05}C_{0.95})CO$ and $BS(G_{0.10}C_{0.90})CO$ samples are shown in Table 2. While the sample particle size distribution of $BS(G_{0.05}C_{0.95})CO$ and $BS(G_{0.10}C_{0.90})CO$ are shown in Figures 7a and 7b. Table 2 shows that the particle size of the Scherrer calculation is greater than the result of the ImageJ software. This is caused by the calculation of ImageJ software only based on the results of SEM characterization . Whereas SEM results are obtained from the observations at one or more point of a particular sample.

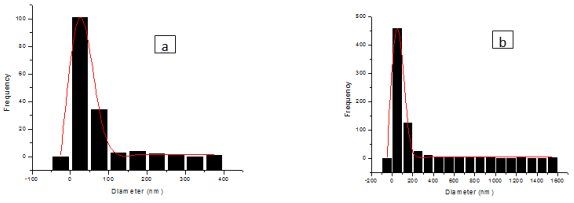

Figure 7. The particle size distribution in the a) $BS(G_{0.05}C_{0.95})CO$ and b) $BS(G_{0.10}C_{0.90})CO$

Table 2. Results of particle size calculation of $Bi_2Sr_2(Ca_{2-x}Gd_xCu_2O_z)$ sample

No	Sample	Particle Size (nm)	
		Scherrer equation	ImageJ software
1	$Bi_2Sr_2(Ca_{0.95}Gd_{0.05}Cu_2Oz)$	30.722	25.345
2	$Bi_2Sr_2(Ca_{0.90}Gd_{0.10}Cu_2Oz)$	55.164	50.321
3	$Bi_2Sr_2(Ca_{0.85}Gd_{0.15}Cu_2Oz)$	71.366	67.985
4	$Bi_2Sr_2(Ca_{0.80}Gd_{0.20}Cu_2Oz)$	94.877	91.423
5	$Bi_2Sr_2(Ca_{0.75}Gd_{0.25}Cu_2Oz)$	101.159	98.291

4. Conclusion

From the results of research that has been done, then it can be concluded:

- The all sample shows a spectrum dominated by high-intensity of superconductor $Bi_2Sr_2(Gd_xCa_{1-x})Cu_2O_z$ and phase impurities of 2201 ($Bi_2Sr_2CuO_x$) and phase 21 (Ca_2CuO_3) with little intensity.
- The addition of the Gd element causes a change of the lattice parameter, so that the unit cell volume becomes larger. In contrast, the addition of the Gd element causes the atomic density in the unit cell to become smaller.
- FTIR analysis yields uptake at 439.77, 632.65, 954.76, 1145.72, 1269.16, 1716.65, 2719.63, 3001.24, 3714.90 and 3844.13 cm-1.
- From SEM characterization results obtained the average particle size ranges from 30-101 nm.

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