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Effect of $CaCO_3$ Levels on Formation Phase Material BSCCO-2212 superconductor with Method Mixing Wet

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Abstract

It has been done a variation of $CaCO_3$, which is 0.95; 1.00; 1.05; and 1,10, through phase formation of BSCCO-2212 superconductor using a wet-mixing method. Samples were dissolved with HNO_3 and distilled by water slowly until the color was clean blue, then gradually dried out at temperatures of 300, 400, and 600 °C. The samples were calcined for 10 hours at 800°C and sintered at 820°C for 20 hours. The XRD's characterization results show that the highest volume fraction was 76.65% in the Ca 1.05 sample. The lowest volume fraction was 49.85% in the Ca 1.10 sample. Meanwhile, the highest degree of orientation was 44.27% in the Ca 1.00 sample, and the lowest degree of orientation was 8.58% in the Ca 1.10 sample. SEM's characterization shows that all samples have been oriented and relatively little space between slabs (voids).

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Abstract

Telah dilakukan penelitian tentang variasi kadar $CaCO_3$ yaitu 0,95; 1,00; 1,05; dan 1,10 mol terhadap pembentukan fase bahan superkonduktor BSCCO-2212 dengan metode pencampuran basah. Sampel dilarutkan dengan HNO_3 dan aquades secara perlahan sampai berwarna biru jernih, kemudian dilakukan pengeringan pada suhu 300, 400 dan 600°C secara bertahap. Sampel dikalsinasi pada suhu 800°C selama 10 jam dan disintering pada suhu 820°C selama 20 jam. Hasil karakterisasi XRD menunjukkan bahwa fraksi volume tertinggi diperoleh sebesar 76,65% pada kadar Ca 1,05 mol. Sedangkan fraksi volume terendah sebesar 49,85% pada kadar Ca 1,10 mol. Sementara nilai derajat orientasi tertinggi sebesar 44,27% pada kadar Ca 1,00 mol dan derajat orientasi terendah sebesar 8,58% pada kadar Ca 1,10 mol. Hasil SEM menunjukkan secara umum semua sampel telah terorientasi dan ruang kosong (*void*) relatif sedikit..

1. Introduction

A superconductor is something material that can deliver current lossless electricity energy because of the temperature low own resistivity worth zero (ρ=0) and induction magnetic worth zero (B = 0). Superconductors were first discovered in 1911 by a Dutch physicist from Leiden University, namely Heike Kamerlingh Onnes. Onnes fabulous mercury (Hg) using liquid helium at a temperature of 4.2K (Darminto et al., 1999).

Superconductor critical high temperature (SKST) is a compound multi-component. It has multiphase, besides characteristic associated anisotropy structure layered and effects fluctuation thermal. One superconductor temperature critical to the vital height is the Bi-Sr-Ca-Cu-O (BSCCO) system. This system is known as 3-phase superconductive, that is, phases of Bi-2201 (Tc ~ 10K), Bi-2212 (Tc ~ 80K), and Bi-2223 (Tc ~ 110K) (Yulianti, 2004). The BSCCO system has a layered structure superconductor, so cause superconductor very BSCCO system fragile and easily broken. It is a superconductor BSCCO system with characteristic high and long anisotropy and short coherence (Herlyn, 2008).

In the BSCCO system, element Ca influences the formation phase and can increase volume fraction. Materials commonly used as a source of Ca in synthesized BSCCO is CaCO3 (Darminto et al., 1999; Cardwell & Ginley, 2002).

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Research conducted previously that influences CaCO3 levels in synthesis superconductor BSCCO-2212 without Pb doping with method reaction solids generated a volume fraction of 78.34% and degrees orientation of 25.6 2%, which is relatively high at CaCO3 levels of $_{1.05}$ (Larasati, 2012). Also, synthesis superconducting Bi-2212 without Pb doping was generated through separate calcination and sintering processes. Calcination and sintering were performed at 800 °C for 10 hours and 820°C for 20 hours (Harnova, 2005).

This research synthesized superconductor BSCCO-2212 with variation CaCO3 levels, namely 0.95; 1.00; 1.05; 1.10 moles using the method of mixing wet. Meanwhile, the temperature and time used for calcination and sintering, respectively, is 800°C for 10 hours and 820°C for 20 hours. Results were then characterized using *X-Ray Diffraction* (XRD) and *Scanning Electron Microscopy* (SEM). As for aspects studied in the formation, the BSCCO-2212 phase is a level purity formed phase with count volume fraction, impurity, and degree orientation.

2. Research Methods

Primary materials used in this research is form material oxides and carbonate with level high purity, namely Bi_2O_3 (99.9 %) of *Stream Chemical*; $SrCO_3$ (99.9 %) of *Stream chemical*; $CaCO_3$ (99.9 %) of *Stream Chemical*; and CuO (99.999 %) of *Merck,* HNO_3 and equates.

The base material is weighed, dissolved with sour nitrate (HNO3) 68%, and slowly distilled water. The material was stirred slowly while heating above *a hot plate* at ± 70°C to solution the colored blue clear, indicating that the solution is homogeneous with pH = 1, then set aside for 24 hours until the crust (Khafifah et al., 2011; Darminto & Widodo, 2010). It is dried at 300, 400, and 600°C in stages for 5, 10, and 20 hours (Marhaendrajaya, 2001). After drying, grind with a *mortar* and *paste* for ± 10 hours (gradually) until the material feels smooth. The sample was pelletized with a *press* with 8 tons of pressure and calcinated at 800 °C for 10 hours after sample-shaped pellets. Calcined samples are imperfect because cavities result from the evaporation of $_{CO2}$ gas, so it needs grinding to repeat for about ± 10 hours. The sample was pelletized again and sintered for 20 hours at 820 °C. Synthesis results superconductor characterized using XRD and SEM. The flow chart study is shown in Figure 1.

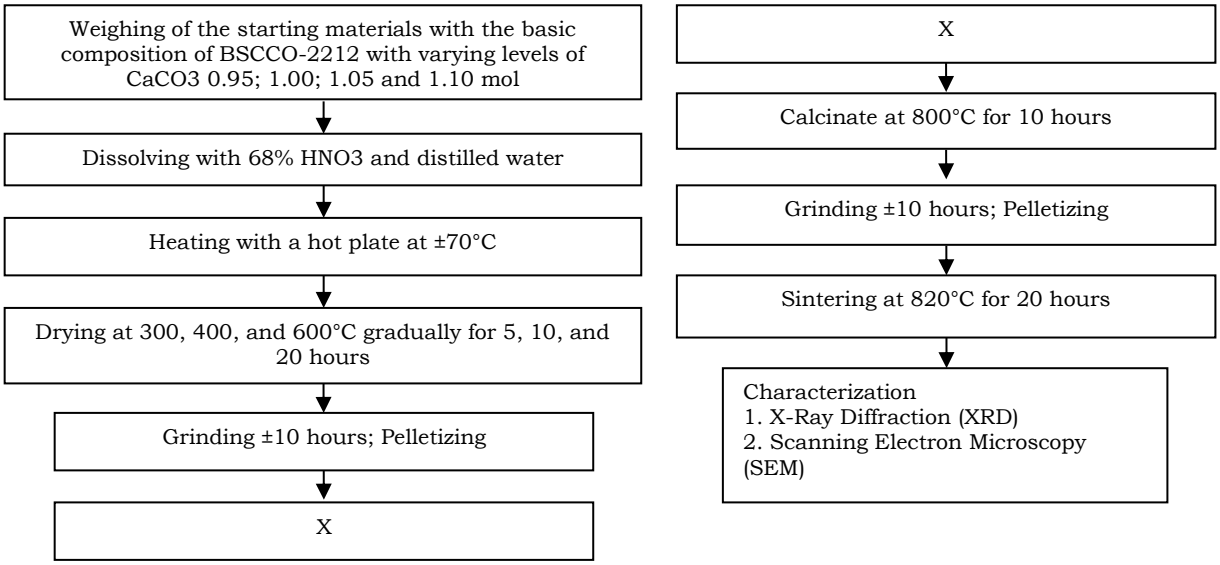

Figure 1. Flow chart study

The purity level of the BSCCO-2212 phase formed was obtained with count volume fraction, impurity, and degree orientation contained within the sample, based on equality (1), (2), and (3).

$$F_V = \frac{\sum I(2212)}{Itotal} \times 100\% \quad \textbf{(1)}$$

$$I = 100\% - F_V \quad \textbf{(2)}$$

$$P = \frac{\sum I(00l)}{\sum I(2212)} \times 100\% \quad \textbf{(3)}$$

F_V is the volume fraction of the BSCCO-2212 phase, I is impurities, and P is degrees orientation. While I (total) is the total intensity, I(2212) is the intensity BSCCO-2212 phase, and I(00l) is the intensity BSCCO-2212 phase with h=k = 0; l number even.

3. Results and Analysis

3.1 Analysis Results in X-Ray Diffraction (XRD)

BSCCO-2212 sample was characterized using X-Ray Diffraction (XRD). The formed diffraction spectrum was analyzed using the *HighScore Plus* program version 3.0e (3.0.5), and the results matched with the ICDD standard. The peaks that appear in the sample are then matched with the method. The phase that appears in the fourth sample can see in **Figure 2**. The phase that appears in the fourth sample can see in **Figure 2**. See the 2θ and d spacing values in the corresponding phase database.

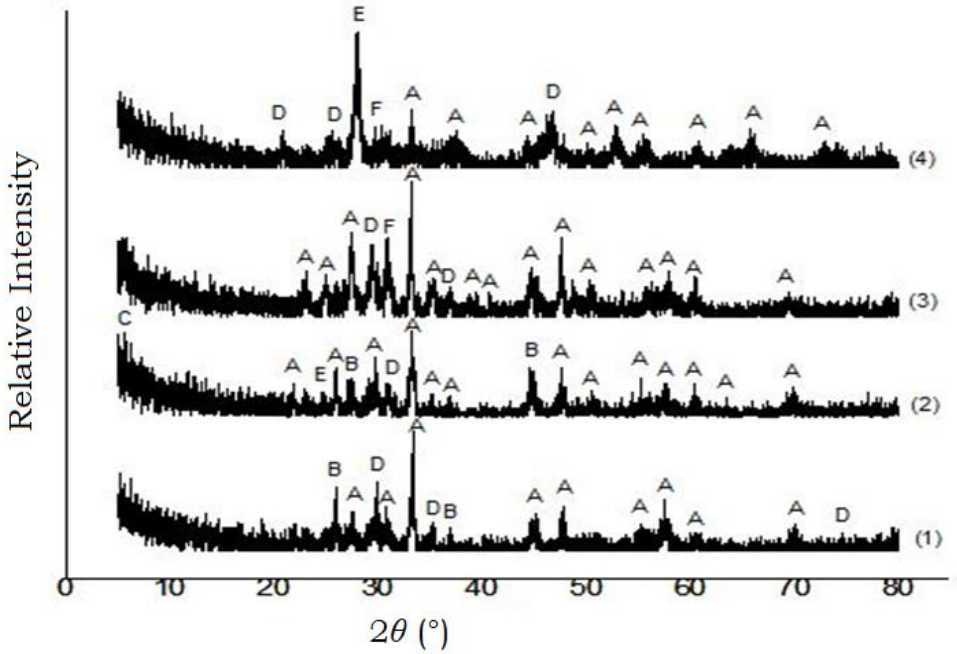

Figure 2. Results of *X-Ray Diffraction* (XRD) analysis (1) BSCCO-2212/Ca 0.95; (2) BSCCO-2212/Ca 1.00; (3) BSCCO- 2212/Ca 1.05; (4) BSCCO-2212/Ca 1.10 mol. (Remarks: (A) BSCCO-2212, (B) BSCCO-2201, (C) BSCCO-2223, (D) $Sr_{1.25}Bi_{0.75}O_{3,}$ (E) $Ca_{0.85}Sr_{0.15}CuO_{2,}$ (F) $Sr_{8}Cu_{13}O_{22.6.}$

Figure 2 shows XRD spectrum on the sample BSCCO-2212/Ca 0.95; BSCCO-2212/Ca 1.00; BSCCO-2212/Ca 1.05; BSCCO-2212/Ca 1.10 mol. The fourth sample succeeds in the BSCCO-2212 phase, although it has yet to form the pure BSCCO-2212 phase. It is marked with appearance peaks BSCCO-2212 phase. In addition, fourth, the sample has also been oriented see Mark P. The highest was BSCCO-2212/Ca 1.00, which was 44.27% obtained from 2θ = 5.5302°hkl (002) and 2θ = 35.168° hkl (00 12), and *the* value *P* Lowest in BSCCO-2212/Ca 1.10 which is 8.58% obtained from 2θ = 60.6924° hkl (00 *20)*.

Based on search-match data results, all phases besides BSCCO-2212 (A) are impurities, including (B), (C), (D), and (E). Appearance phase impurity on the fourth sample is possible due to relatively poor grinding and pelletizing processes (Suprihatin, 2008), so formed unwanted compounds (impurities). _

Based on the results of XRD analysis, volume fraction (*Fv)*, impurity (*I)*, and degree orientation (*P)* are indicated in **Table 1**.

Table 1. Volume fraction, impurities, and degrees of orientation

Sample Code	Volume Fraction (%)	Impurity (%)	Degrees Orientation (%)
BSCCO-2212/Ca 0.95	52,41	47,59	39.95
BSCCO-2212/Ca 1.00	53,66	46,34	44,27
BSCCO-2212/Ca 1.05	76.65	23.95	16.35
BSCCO-2212/Ca 1.10	49.85	50,15	8.58

The addition of CaCO3 levels in the sample can influence the formation phase in the BSCCO system (Ginley et al., 2002). based on **Table 1** on BSCCO-2212/Ca 1.05 obtained highest volume fraction of 76.65%, while BSCCO-2212/Ca 1.10 was obtained lowest volume fraction of 49.85%. In addition, BSCCO-2212/Ca 0.95 obtained a mark volume fraction of 52.41%, and BSCCO-2212/Ca 1.00 had a mark volume fraction of 53.66%. The connection between variation CaCO3 levels to level purity BSCCO-2212 phase includes volume fraction (*Fv)*, impurities (*I)*, and degrees orientation (*P)* can see in **Figure 3**.

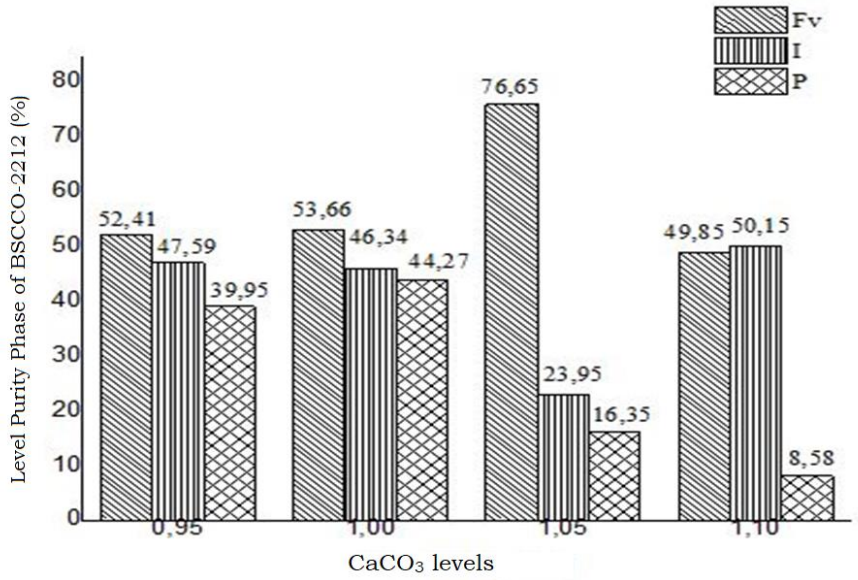

Figure 3. Connection variation $CaCO_3$ levels with level purity BSCCO-2212 phase.

Figure 3 shows that the volume fraction increased at BSCCO-2212/Ca 0.95 to BSCCO-2212/Ca 1.05, then decreased from BSCCO-2212/Ca 1.05 to BSCCO-2212/Ca 1.10. Degrees orientation experience enhancement from BSCCO-2212/Ca 0.95 to BSCCO-2212/Ca 1.00, then decreased at BSCCO-2212/Ca 1.00 to BSCCO-2212/Ca 1.10.

3.2 Results of *Scanning Electron Microscopy* (SEM) Analysis

Analysis results SEM BSCCO-2212 at BSCCO-2212/Ca 0.95; BSCCO-2212/Ca 1.00; BSCCO-2212/Ca 1.05; and BSCCO-2212/Ca 1.10 with 2000x magnification can seen in **Figure 4**.

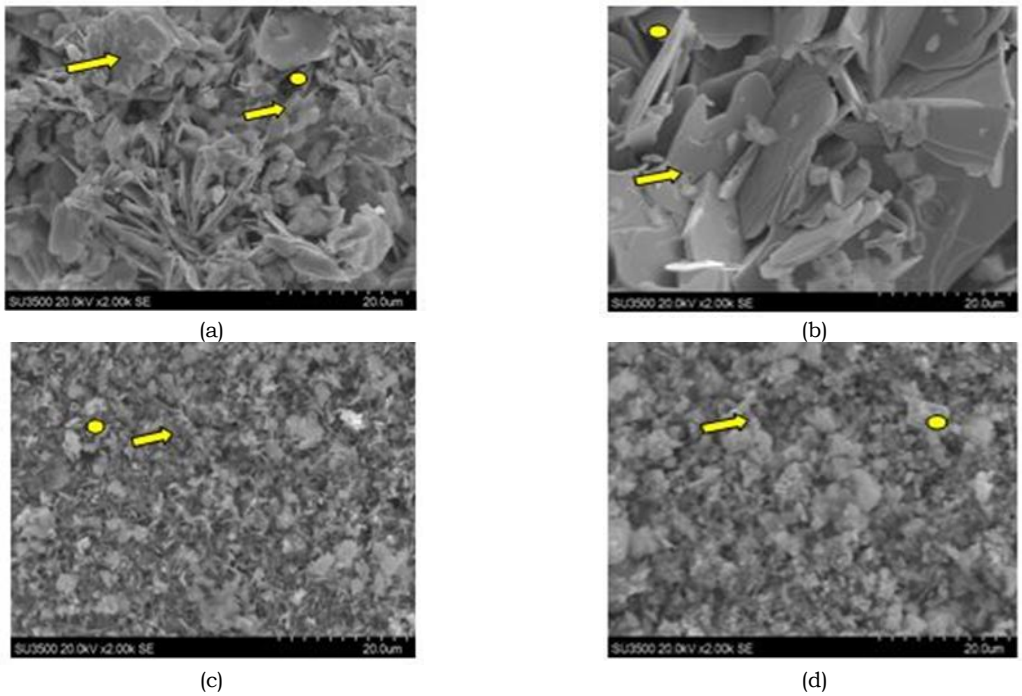

Figure 4. SEM analysis results on the sample superconductors (a) BSCCO-2212/Ca 0.95; (b) BSCCO-2212/Ca 1.00; (c) BSCCO-2212/Ca 1.05; (d) BSCCO-2212/Ca 1.10 mol. * Description: ( ) slabs that have oriented, and ( ) space empty (*void)*, * magnification 2000x.

Based on **Figure 4** can see that the formation structure fourth crystal sample has shown polished plates unidirectional or already oriented with relative voids little. Formation crystal best with the best P in the BSCCO-2212/Ca 1.00 sample of 44.27%.

4. Conclusions

Based on the research that has been done, the addition of CaCO3 levels significantly affects the purity of the BSCCO-22 12 superconducting phase. At BSCCO-2212/Ca 1.05, the highest volume fraction (Fv) was obtained, 76.65 %, and the lowest volume fraction was found in BSCCO-2212/Ca 1.10, i.e., 49.85%. Degrees the highest orientation (P) at BSCCO-2212/Ca 1.00, i.e., 44.27%, and the lowest at BSCCO-2212/Ca 1.10, i.e., by 8.58%. SEM characterization results show that microstructure material BSCCO-2212 superconductor has oriented and own room relatively empty (*void)* f little.

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