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Effect of Sintering Temperature on BPSCCO Superconductor with Addition of Al_2O_3 Using Wet Mixing Method

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Abstract
In this work, the synthesis of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ (BPSCCO) superconductors added with Al_2O_3 nanoparticles has been investigated. Al_2O_3 nanoparticles have a function to form a phase so that it can increase the superconductivity properties of BPSCCO. BPSCCO added with Al_2O_3 nanoparticles was synthesized using the wet mixing method with variations in sintering temperature (840 and 880°C). The performance of BPSCCO with the addition of Al_2O_3 was carried out by testing XRD, SEM, and I-V. Based on the results of the XRD characterization, the volume fraction, impurity, and crystal size obtained were 57.49% (BPSCCO-840), 38.96% (BPSCCO-880); 42.5% (BPSCCO-840), 38.96% (BPSCCO-880) and 21.38 nm (BPSCCO-840); 28.84 nm (BPSCCO-880). Based on the results of the SEM test, the sample plates are regular and have a small empty space. Based on the results of the I-V test, the BPSCCO-840 sample has the highest electrical conductivity of 2.4x104 S/m at a current of 0.6 mA and the BPSCCO-880 sample has an electrical conductivity of 2.8x104 S/m at a current of 0.3 mA.
Keywords: Superconductor; Al_2O_3; BPSCCO; Wet Mixing Method.

1. Introduction

Superconductors are materials that can conduct electric current without resistance [1]. Along with the development of superconducting technology, it is increasingly being used, for example in the railway sector, namely super-fast trains known as Magnetic Levitation (MagLev) and the manufacture of electromagnets [2]. In the medical field for the manufacture of Magnetic Resonance Imaging (MRI) diagnostic tools [3]. The phenomenon of superconductivity is observed under special conditions, when it enters a certain temperature, the resistivity is immediately zero [4]. These conditions provide convenience in delivering current that enters the superconducting temperature criteria. The temperature at which superconductivity events occur is called the transition temperature or critical temperature (T_c), where a material is in a transition phase, namely from conditions that have normal electrical resistance to superconductivity conditions [5], [6].

The obstacle that is still faced in the application of superconductors is the superconductivity of materials that appear at low temperatures ($T_c < 0^oC$) [4]. Efforts to increase the critical temperature and reduce the brittleness of the material are overcome by bismuth (Bi)-based superconductors, known as the Bi-Sr-Ca-Cu-O system or bismuth strontium calcium copper oxide (BSCCO) [7]. The BSCCO superconductor has three phases with the composition $Bi_2Sr_2Ca_{n-1}CunO_{2n+4+\delta}$ with n = 1, 2, 3, namely phases 2201 (T_c=12 K), 2212 (T_c=95 K), and 2223 (T_c=105 K). Among the three phases, Bi-2223 has the highest transition temperature over liquid nitrogen and is a type II superconductor. Pb doping on Bi-2223 (BPSCCO) can be done to increase the stability of the structure and increase Tc (~110K) [7]. In practice, BPSCCO has a higher magnetic field than conventional sensors and is easy to manufacture. The disadvantage of BPSCCO is the current density (J_c) which is quite low and easily drops when given an external field at high temperatures. The addition of nanometer-sized particles such as Al_2O_3, Fe_3O_4, SnO_2, and NiO can increase the pinning flux, critical current density (J_c), and phase formation [8].

The method that is often used to synthesize BPSCCO superconductors is the solid state method because the process is simple. However, this method has the disadvantage of low homogeneity and requires a long heat treatment. Another synthesis method that can be used to synthesize BPSCCO superconductors is the wet mixing method. This method is used because it has the advantages of being easy to make and simple and inexpensive in synthesizing superconducting materials [9]. The synthesis of BPSCCO superconductors using the wet mixing method can produce high homogeneity and can produce superconductors with good quality [11]. The effect of heat treatment on the calcination and sintering process of BPSCCO synthesis has an effect on the process of reducing superconducting impurities and increasing the critical temperature value (T_c). In addition, heat treatment is also used to optimize crystal formation.

In this research, the synthesis of BPSCCO by doping Al2O3 nanoparticles by varying the sintering temperature (840 and 880°C) using the wet mixing method will be carried out. The addition of Al_2O_3 nanoparticles is used to improve superconducting properties, one of which is the critical current density and lowers the critical temperature [10]. The results obtained were characterized by X-Ray Diffraction (XRD) to determine the level of phase purity, Scanning Electron Microscopy (SEM) to determine the microstructure of the sample, and Tc test to determine the critical temperature of the sample.

2. Method

The materials used in this study include Bi_2O_3 98%, $CaCO_3$ 98%, PbO_2 97%, $SrCO_3$ 96%, CuO 99%, Al_2O_3, HNO_3 68%, HCl, and distilled water. All precursors have a calculation to determine the mass of each precursor using the equation:

$$0.8Bi_2O_3+0.4PbO_2+2SrCO_3+2CaCO_3+3CuO \rightarrow Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}+4CO_2 \quad \ldots\ldots 1)$$

Where the mass of Bi_2O_3 is 1.09286 g, $CaCO_3$ is 0.58686 g, PbO_2 is 0.28051 g, $SrCO_3$ is 0.86559 g, and CuO is 0.69962 g. The mass ratio of Al_2O_3 and BPSCCO is 1:3. Meanwhile the tools used are an analytical balance, furnace, agate mortar, spatula, 12 mm diameter pellet mold, magnetic stirrer, 200 mL and 500 mL beakers, and a press machine. Sterilization of tools using acetone solution. The synthesized started by weighing all superconducting materials according to stoichiometric calculations. Then dissolve 98% $CaCO_3$ powder, 97% PbO_2, 96% $SrCO_3$, 99% CuO, and Al_2O_3 powder each in 10 mL distilled water, called solution A. After that, dissolve 98% Bi2O3 powder into 20 mL of HNO_3 solution, called solution B. Mix solution A and solution B and stir using a magnetic stirrer. Heating the mixture using a hot plate at a temperature of about 70°C until the solution boils. Calcining the sample at 600°C for 40 hours. Grind the sample followed by pelletization. Then sintering the sample at variations of 840 and 880°C for 10 hours with heating rate 5°C/min. Structural and morphological properties of BPSCCO-Al_2O_3 superconducting materials were characterized using XRD and SEM. Meanwhile, the superconductivity is characterized using the 4-probe method.

3. Result and Discussion

3.1. X-ray Diffraction Analysis

The results of XRD characterization were used to determine the crystal structure formed in the sample. The data obtained from the X-ray diffraction process of Cu-Kα with a wavelength of 1.5406 Å. Based on Figure 1, it shows a graph of the relationship between 2θ and the intensity of the BPSCCO- Al_2O_3 sample which was sintered at 840°C and 880°C. There are 3 types of superconducting materials which are indicated by the graph peaks at a certain 2θ position. The superconducting materials contained in the sample are Bi-2223, Bi-2212, and Bi-2201. BPSCCO 2223 is the most optimal phase for superconducting applications because it has a critical temperature of 110 K [11]. The peak position of the material results from the phase matching of the sample. Based on the results of the XRD characterization, shows that the sample has formed the BPSCCO 2223 phase. This is indicated by the formation of the BPSCCO 2223 phase. The BPSCCO 2223 sample is indicated by the presence of a peak that has an index h=k=0 and l=even number [12]. In Figure 4, sample 880 has more BPSCCO 2223 peaks than sample 840. The peak intensity of sample 880 is also higher than sample 840. The addition of Al_2O_3 nanoparticles plays a role in increasing the BPSCCO 2223 phase. The optimum sintering temperature is produced at a temperature below the partial melting temperature [13]. The partial melting point decreased with the addition of Al_2O_3 nanoparticles there by increasing the number of BPSCCO 2223 phases in the sample sintered at 840 and increased at 880. In sample 840 there were more BPSCCO 2212 peaks. The peak intensity of BPSCCO 2212 was higher in sample 840. Both samples showed sharp peaks dominated by the impurity phase. The peaks formed indicate poor crystallinity.

Based on Table 1, it is known that the sintering temperature of the BPSCCO- Al2O3 material causes a decrease in the volume fraction which is in accordance with the research conducted by Lusiana [14]. The increase in sintering temperature caused the sample impurity to increase from 42.50% to 61.04%. The formation of impurities depends on the sample depending on the initial molar composition of the ingredients that make up BPSCCO. Sample impurity is an imperfect phase in the formation of BPSCCO material. Impurities are characterized by the formation of peaks as a result of XRD characterization which results in amorphous forms and low peak intensity [15].

3.2. Scanning Electron Microscope Analysis

SEM characterization results showed the surface morphology of BPSCCO- Al_2O_3 samples at sintering temperatures of 840^{o}C and 880^{o}C. There are differences in the surface morphology of the samples with sintering temperatures of 840 and 880^{o}C. Based on Figure 2, it can be seen that there is little BPSCCO 2223 material at a sintering temperature of 840^{o}C because it has not been completely formed. In samples with a sintering temperature of 880^{o}C, BPSCCO-2223 material was formed but not completely dispersed. Figures 2a and 2b show the surface of sample 840 in the form of grains and there are planar plates which indicate the presence of BPSCCO-2223 superconductors. The presence of space between the grains of the compound indicates the porosity of sample 840. Figure 2c shows the results of the SEM characterization of sample 880, a BPSCCO-2223 phase is formed which is marked with plates directed in a certain direction. Based on the results of SEM characterization, it appears that the increase in sintering temperature causes the degree of orientation to increase. At a sintering temperature of 880^{o}C, it has less porosity, increased planar structure, and decreased fine grain formation [16]. The increase in sintering temperature causes the degree of organization of the BPSCCO-2223 plate to increase. The addition of Al_2O_3 nanoparticles affects increasing the formation of the BPSCCO-2223 phase. Al_2O_3 nanoparticles tend to be stable at temperatures below the partial melting point (<900^{o}C) so that at 880^{o}C Al_2O_3 nanoparticles are easier to imagine forming the BPSCCO-2223 phase [17]. At 840^{o}C, the Al_2O_3 nanoparticles are stable and tend not to change morphologically.

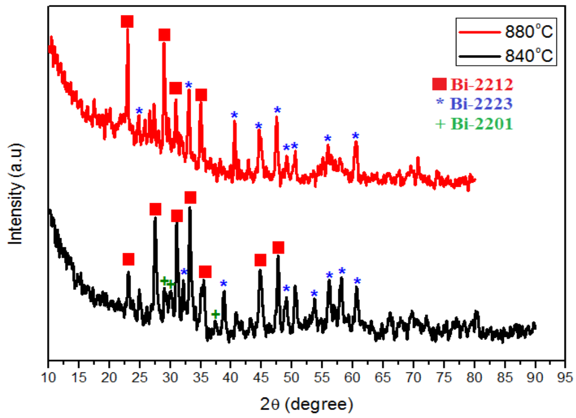

Figure 1. The result of XRD characterization of BPSCCO with Al_2O_3 addition at variation of sintering temperature of 840^{o}C and 880^{o}C.

Tabel 1. Calculation Result of Impurity and Crystallite Size of BPSCCO-Al_2O_3.

No.	Sample Name	Volume Fraction (%)	Impurity (%)	Crystallite size (nm)
1	840	57.50	42.50	21.38
2	880	38.96	61.04	28.84

3.3. I-V Analysis

I-V analysis was carried out using the 4-probe method in order to obtain the current and voltage relationship graph shown in Figure 3. Based on Figure 3, the pattern of voltage spikes shows a transition from the superconducting state to the conductor state. This is because the conductivity measurement was carried out at room temperature [18].

Based on Figure 4, shows the results of the I-V test characterization which displays a graph of the relationship between current and electrical conductivity. I-V characterization was carried out at room temperature. The electrical conductivity tends to increase as the sintering temperature increases. The sample with a sintering temperature of 840°C showed an increase in electrical conductivity with the addition of current. In contrast to the sample with a sintering temperature of 880°C which showed an increase in electrical conductivity to a current of 0.3 mA, then decreased with increasing current.

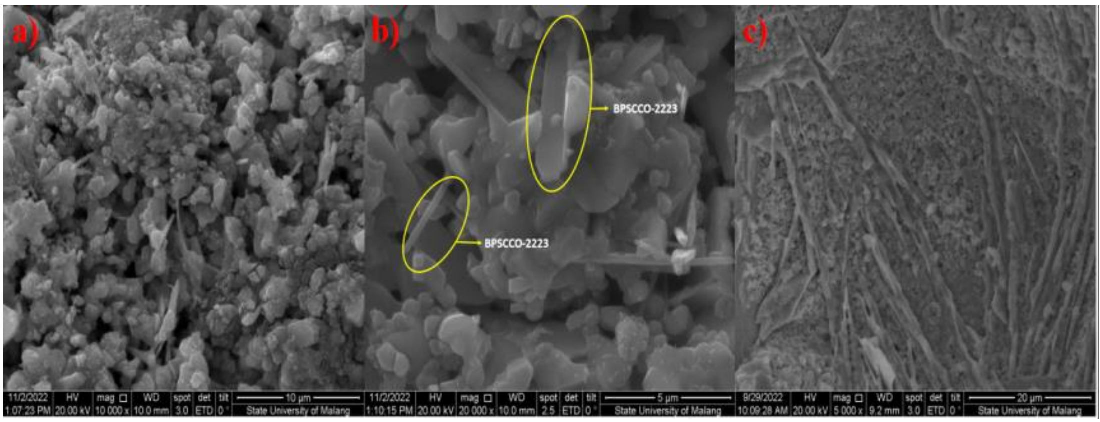

Figure 2. The result of SEM characterization of BPSCCO with Al_2O_3 addition at variation of sintering temperature of a) 840°C at 10 µm magnification, b) 840°C at 5 µm magnification, and c) 880°C at 20 µm magnification.

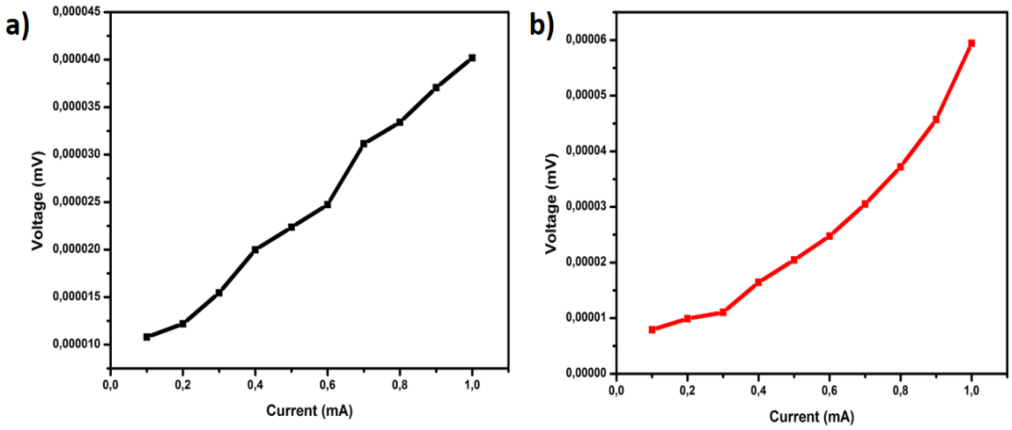

Figure 3. The result of I-V characterization of BPSCCO with Al_2O_3 addition at variation of sintering temperature of a) 840°C and b) 880°C.

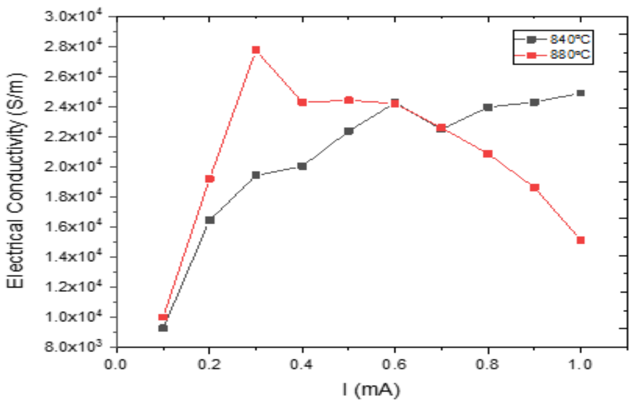

Figure 4. Graph of electric current and conductivity of BPSCCO with Al_2O_3 addition at variation of sintering temperature of a) 840°C and b) 880°C.

4. Conclusion

BPSCCO added with Al_2O_3 nanoparticles has been successfully synthesized using the wet mixing method. Based on the results of the XRD characterization, the volume fraction, impurity, and crystal size obtained were 57.49% (BPSCCO-840), 38.96% (BPSCCO-880); 42.5% (BPSCCO-840), 38.96% (BPSCCO-880) and 21.38 nm (BPSCCO-840); 28.84 nm (BPSCCO-880). Based on the results of the SEM test, the sample plates are regular and have a small space. The addition of Al_2O_3 to the BPSCCO-840 and BPSCCO-880 samples increased the number of BPSCCO 2223 phases. The formation of the BPSCCO 2223 phase was optimum at sintering temperature 880^{o}C because this temperature is close to the partial melting temperature for BPSCCO superconducting materials. Based on the results of the I-V test, the BPSCCO-840 sample has the highest electrical conductivity of 2.4x104 S/m at a current of 0.6 mA and the BPSCCO-880 sample has an electrical conductivity of 2.8x104 S/m at a current of 0.3 mA.

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