J. Sol. State Sci. Technol. Letts. Vol. 22 No. 1 & 2 (2021): 12-17
http://myjms.mohe.gov.my/index.php/jssstl

Effect of Different Sintering Condition on $Bi_2Sr_2Ca_2Cu_3O_x$ (Bi-2223) Superconductors Prepared via Co-Precipitation Method

S. Nabilah Abdullah[1], M. M. Awang Kechik[1,*], S. A. Halim[1], S. K Chen[1], K. P. Lim[2]

Superconductor and Thin Film Laboratory, Department of Physics,
Faculty of Science, Universiti Putra Malaysia,
43400 UPM Serdang, Selangor, Malaysia

*****Corresponding author: mmak@upm.edu.my

Abstract: This paper investigates the ability of high temperature superconductor of bismuth-strontium-calcium-copper-oxide $Bi_2Sr_2Ca_2Cu_3O_x$ (Bi-2223) bulks with effect of the oxygen content. The Bi-2223 powders were prepared by using co-precipitation method in order to get a good homogeneity and ultrafine BSCCO powder. The good precursor powder was produced in different sintering times which are 24 and 48 hours at sintering temperature of 850 °C. The samples were divided into two different sintering condition which is sintering; flowing the oxygen through the furnace and without flow of oxygen. The phase, structure andsuperconducting properties of the samples were analysed by X-ray diffraction (XRD) and AC Susceptometer. The result showed that the samples with sintered by flowing of oxygen was increased the critical temperature (T_c) with the better grain alignment. XRD analysis also showed a good percentage of the Bi-2223 as a major phase and Bi-2212 as a secondary phase for all samples.

Keywords: BSCCO; Superconductor; Co-precipitation; Critical temperature (T_c)

Superconductor is material that have an ability of conducting electricity or transport electrons from one atom to another atoms with no losses of any kind. A substance become superconducting where the resistance become zero value and this case usually happens at very low temperature or very cold often as low as 10K or -273°C. The first observed and discovered in 1911 by a Dutch physicist named Heike Kamerlingh Onnes. He came out with the fact that resistance become zero as known as superconducting state. There are many application of superconductor such as were used in electric power, transportation, Medical Imaging and Diagnostics, industrial processing, wireless communication, instrumentation, sensors, standard and radar, high-end computing and NMR (Nuclear Magnetic Resonance). The addition of Ca to this discover of bulk superconductivity at 85K and evidence of superconductivity at 110K in the Bi-Sr-Ca-Cu-O system [7]. There are three phases of general formula for $Bi_2Sr_2Ca_{n-2}Cu_nO_x$ with n=1,2 and 3 exist which are closely related from structural point of view [8]. The structures are pseudo tetragonal that can be described as a stacking of basic $Bi_2Sr_2CuO_6$ unit with either zero, one or two $CaCuO_2$ slabs inserted. The phases actually referred for 2201, 2212 and 2223 with the respect to the increasing values of n and this number represents the cation's numbers for each element in the unit cell. The number of Cu-O planes actually related to T_c and it will increase when the number of Cu-O layers increase which is n up to n=3 [2]. For Bi-2201, it critical temperature, T_c is approximate to 20 K, for Bi-2212, it critical temperature, T_c is approximate to 85K and for Bi-2223, it critical temperature, T_c is approximate to 110 K [1]. BSCCO is one of important category of high temperature superconductors which is it did not contain rare element and shares a two dimensional layered as known as perovskite structure where the phenomenon of superconducting differ in the plane of copper oxide. Compare to YBCO, BSCCO gives the high T_c value and it more stable in superconducting behaviour with

respect to oxygen loss. BSCCO compound shows both which are an isotropic or dimensional behaviour and an intrinsic Josephson Effect [6]. The Bi-2223 phase is known as the high T_c phase which reach at zero resistance at about 110 K. This phase is very difficult to obtain in bulk material with a high proportion. Certain dopants, usually Pb have been found to promote the formation of the Bi-2223 phase. The functions of Pb more likely to decrease the melting point of Bi compound and increase the range of the temperature for annealing and improve the formation from 2212 phase to 2223 phase [2].is This work are focused to synthesize a good purity $(Bi_{1.6}Pb_{0.4})Sr_2Ca_2Cu_3O_{10}$ (Bi-2223) precursors with fine grain by using co-precipitation method, to investigate the effect of oxygen content in the formation of Bi-2223 bulk and to compare the critical temperature (T_c) and lattice parameter of Bi-2223 material with flow of oxygen and without flow of oxygen.

There are several methods to prepare high T_c superconductor of Bi-2223 such as solid state and wet techniques (sol-gel method, thermal treatment method and co-precipitation method). But in this case, the preparation of Bi-2223 by using co-precipitation method which is in wet method categories [3] were used. This method were chosen to get good homogeneity, to make low reaction of temperature, to produce finer sample and to make sure that the sample in uniform particle size [10]. Otherwise this method is one of the ways to overcome the problem of the difficulty to get the single phase of Bi-2223 [3].

The metal compound such as Bismuth acetate Bi(OOCCH3)3, Lead(II) acetate Trihydrate Pb(CH3COO)2 · 3H2O, Strontium acetate Sr(OOCCH3)2 · 0.5H2O, Calcium acetate Ca(OOCCH3)2 · XH2O, Copper(II) acetate monohydrate Cu(OOCCH3)2 · H2O was measured by using electronic balance machine and then mix with 500ml of acetate acid labelled as Solution A. The solution was stirred in 400 rpm in 80°C. After the mixture homogenous Solution A was placed in the ice bath and continuously stirred in 400 rpm to prevent solution from crystallize. For the Solution B, 300 ml of propan-2-ol was added with 200 ml distilled water and label as Solution B. After that, 25 g of oxalic acid was added and stirred in 400 rpm without heating. After the solution is homogenous, the beaker was soaking into ice bath and continue stirred. Solution A and B was mixed together after both were solute and achieved the concentration of 0.5 M at the range of 0°C to 2°C in the ice bath. After the milky blue solution was formed, the solution was filtered by using Buchner funnel, Buchner flask, vacuum, filter paper with diameter 240 mm before dry on hot plate of 80°C for 12 hours to make sure that sample is totally dry. Then, the sample was ready for pre-calcination at 730°C for 12 hours and for second calcination at 730°C for 24 hours. The purpose of calcination is to produce pure powders by eliminating the undesirable compositions. Next, the sample were pelletized by using 12 ton hydraulic press before undergo the sintering process in the tube furnace at 850°C for 48 hours. For the first batch, the some of the pallets were sintered by flowing the oxygen through the furnace. For the second batch, other pellets were sintered in the open air or without flow of oxygen. The samples were undergo characterization by using x-ray diffraction (XRD) and AC Susceptometer.

X-ray diffraction (XRD) patterns of Bi-2223 for different sintering process with the oxygen flow and in the open air are shown in the Figure 1. The results indicate that Bi-2223 phase is the dominant phase while Bi-2212 phase was detected as a minor phase in the samples sinter with the flow of oxygen and without flow of oxygen or in the open air. The appearance of more than two phases could be related to the stacking faults along the c-axis [5]. The crystal structure for all samples were confirmed to be tetragonal for the value obtained had fulfilled the general equation of tetragonal crystalline structure for ‘a’ equals to ‘b’ but not equal to ‘c’ lattice parameter ($a = b \neq c$) as shown in the Table 1. There also was existence of high-Tc

phases which is Bi-2223 and low Tc phases which is Bi-2212 in the XRD graph for both sintering method. The peak for Bi-2223 was labelled as H (hkl) and for Bi-2212 phase was labelled as L (hkl) as referred to the standard Miller Indices. All the peaks were obtained in the range of 20° > 2θ > 80°.

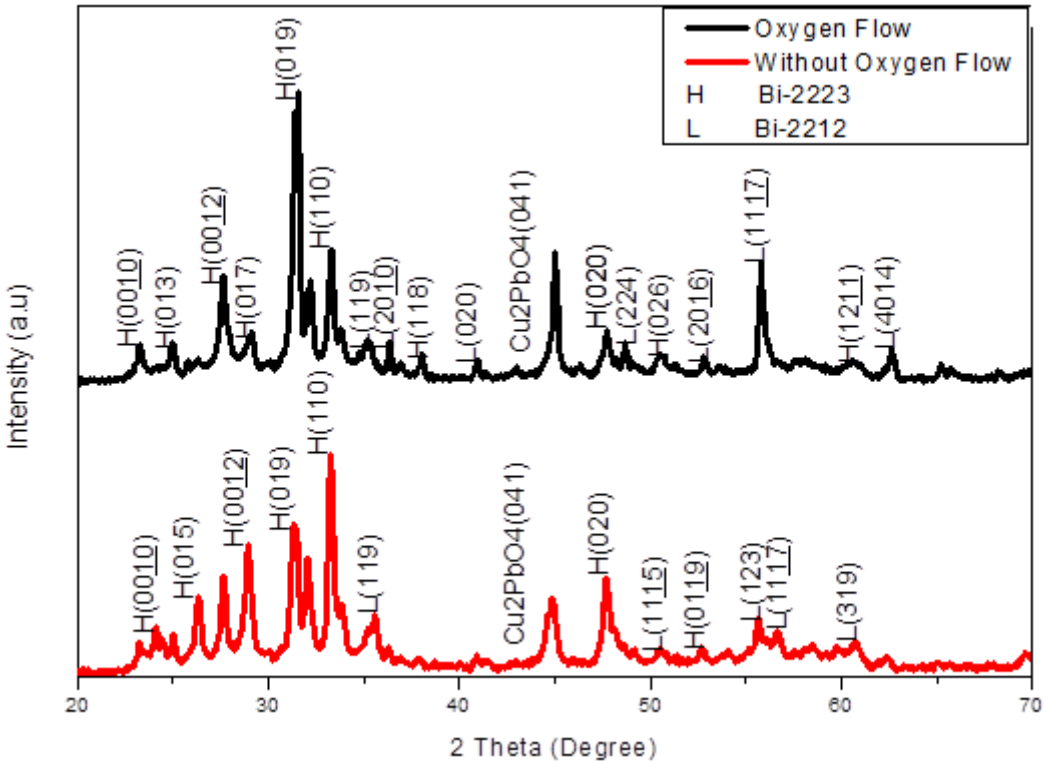

Figure 1. XRD pattern of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ with different sintering method (flow of oxygen and without oxygen flow (in open air) at 850°C). Black line represents sintering process with oxygen flow while red line represents sintering process in open air. Bi-2223 labelled as H (*hkl*) while Bi-2212 labelled as L (*hkl*).

Table 2 shows the volume fraction for all the samples Bi-2223 at different sintering in oxygen flow and in open air**.** From Table 2, the results shown that oxygen flow on the sintering time can increase the volume fraction of phase formation of Bi-2223 phase. The volume fraction of phase formation Bi-2223 phase increased from 61.8 % when sintering process in open air to 74.4 % when sintering process with flow of oxygen. On the other hand, the volume fraction of phase formation Bi-2212 phase decreased from 32.8 % for sintering process in the open air to 1.8% for sintering process with the flow of oxygen. However, the extra peaks belong to impurities that were observed in both samples. These impurities phases were identified as Cu_2PbO_4. The formation of impurities of Cu_2PbO_4 is due to incomplete transformation of Bi-2212 [2]. Table 2 showed the volume fraction of phase formation of impurities Cu_2PbO_4 for sintered without oxygen flow is 5.4% and the impurities were increased for sintered with oxygen flow which is 23.8%.

Table 1. The lattice parameter the samples that sintering with flow of oxygen and sintering in open air

Sample	Lattice Parameter			Volume of cell
	a (Å)	b(Å)	c (Å)	(V / 10^6 pm^3)
Bi-2223 (without oxygen flow)	3.828	3.828	37.150	544.386
Bi-2223 (with oxygen flow)	3.814	3.814	38.307	557.184

Table 2. The volume fraction for all the samples Bi-2223 at different sintering in oxygen flow and in open air

Sample	Volume Fraction of Phase Formation (%)		
	Bi-2223	Bi-2212	Cu_2PbO_4
Bi-2223 (without oxygen flow)	61.8	32.8	5.4
Bi-2223 (with oxygen flow)	74.4	1.8	23.8

The normalized (a.u) versus temperature (K) curves for the samples of Bi-2223 which were subjected to treatment of sintering at 850°C with oxygen flow and sintering at 850°C without oxygen flow or in the open air are shown in the Figure 2 and Figure 3. Based on the graph of Bi-2223 sintered without oxygen flow, the curves shows that it has double transition which indicate there might exist another phase and the possible phase is Bi-2212. The production of high quality superconductor tapes is the presence of secondary phases, such as alkaline earth cuprates or Bi-2212 [4]. It was found from the others research, the double transition occurs because of impurities that exist in the sample that disturb the superconducting element inside the grain. These secondary phases tend to reside on grain boundaries and even in small amounts, they could dominate the transport properties of the tapes [9]. For sintered without oxygen flow, Tc-onset is 109.04K and for sintered with oxygen flow, Tc-onset increase to 110.19K as shown in the Figure 3.

.

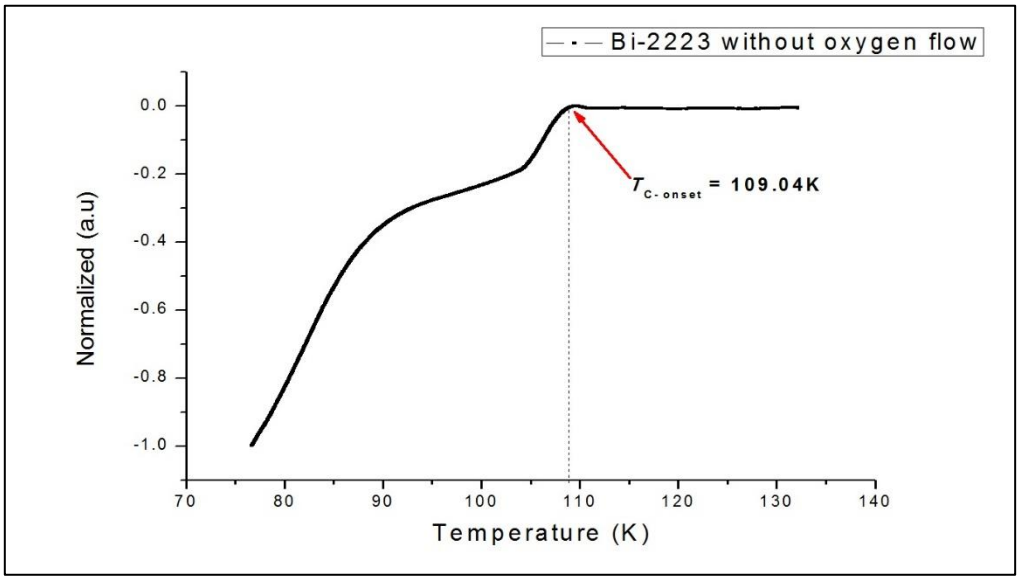

Figure 2. Graph of the resistance versus temperature of Bi-2223 sintered at 850°C in open air labelled with *T*c-onset

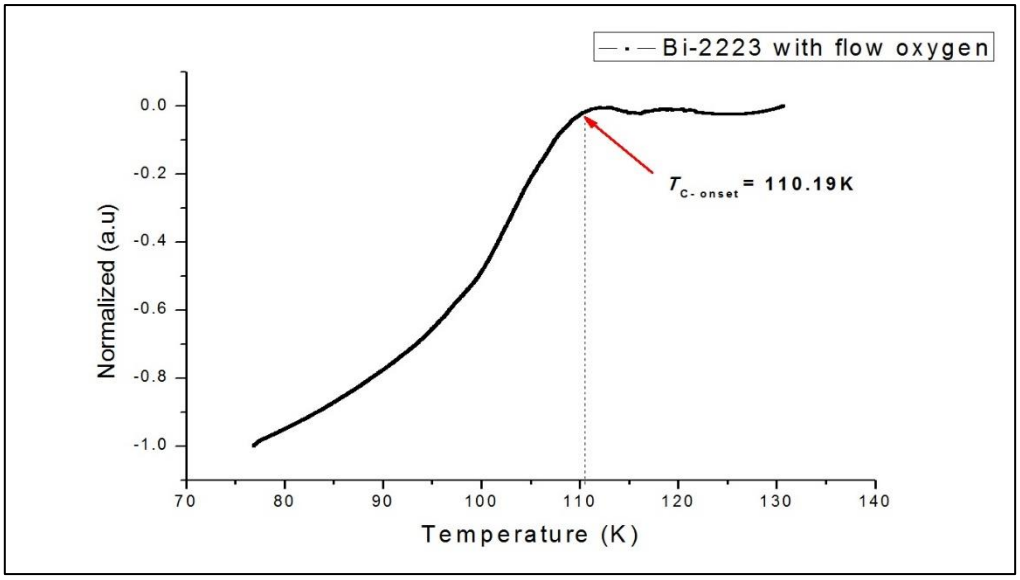

Figure 3. Graph of the resistance versus temperature of Bi-2223 sintered at 850°C in flow of oxygen labelled with *T*c-onset

In conclusion, **s**amples of powder Bi-2223 had been successfully prepared via co-precipitation method. The results that were observed from XRD analysis showed a good percentage of the Bi-2223 for all samples. The calculated lattice parameters of the samples showed that all the samples are nearly similar, where a = b but ≠ c. The volume fraction of phase formation Bi-2223 phase is higher but the volume fraction of phase formation Bi-2212 phase is less when sintered with flow of oxygen. On the other hand, the volume fraction of phase formation of impurities Cu_2PbO_4 is higher for sintered with oxygen flow. From ACS result shows that different sintering condition which is sintered with flow of oxygen and without flow of oxygen gives different type of graph for normalized versus temperature. The graph for sintered without oxygen flow showed the second transition graph. For sintered without oxygen flow, $T_{c\text{-onset}}$ is 109.04K and when samples sintered with oxygen flow, $T_{c\text{-onset}}$ increase to 110.19 K.

Acknowledgement: The authors are grateful for the financial support given by the Universiti Putra Malaysia for the scholarship under Graduate Research Fellowship (GRF) and this work also was supported by superconducting group of Physics Department of Universiti Putra Malaysia with grant number 5540036.

References

[1]. Boussouf, N., Mosbah, M. F., Bouaicha, F., Amira, A., (2012). Effect of magnesium on the Bi-based (2212) superconductors. *Intentional Conference on Electronic, Biomedical Engineering and Its Application (ICEBEA)*, 7-8.

[2]. Guldeste, A. (1994). *Bismuth based thin film superconductors* (Doctoral dissertation, University of Oxford).

[3]. Hamadneh, I., Halim, S. A. & Lee, C. K. (2006). Characterization of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ ceramic superconductor prepared via co-precipitation method at difference sintering time. *Journal of Materials Science* 41: 5526-5530.

[4]. Ismail, M., Abd-Shukor, R., Hamadneh, I., & Halim, S. A. (2004). Transport current density of Ag-sheathed superconductor tapes using Bi-Sr-Ca-Cu-O powders prepared by the co-precipitation method. *Journal of materials science*, *39*(10), 3517-3519.

[5]. Jabur, A. R. (2012). B2223 High Temperature Superconductor wires in silver sheath, Filament diameter effect on critical temperature and current density. *Energy Procedia*, *18*, 254-264.

[6]. Kanungo, S. (2013). Synthesis and Characterization of Gd doped BSCCO-2212. India: National Institute of Technology Rourkela.

[7]. Maeda, H., Tanaka, Y., Fukutomi, M. & Asano, T. (1988). A new high-Tc oxide superconductor without a rare earth element. *Japanese Journal of Applied Physics*, 27, L209.

[8]. Tarascon, J. M., McKinnon, W. R., Barboux, P., Hwang, D. M., Bagley, B. G., Greene, L. H., Hull, G. W., LePage, Y., Staffel, N., & Giroud, M. (1988). Preparation, structure, and properties of the superconducting compound series Bi 2 Sr 2 Ca n− 1 Cu n O y with n= 1, 2, and 3. *Physical Review B*, *38*(13), 8885.

[9]. Umezawa, A., Feng, Y., Edelman, H.S., High, Y.E., Larbalestier, D.C., Sung, Y.S., Hellstrom, E.E., Fleshler, S. (1992). Electromagnetic granularity, critical current density, and low-Tc phase formation at the grain boundaries in (Bi,Pb)2Sr2Ca2Cu3Oχ silver-sheathed tapes, *In Physica C: Superconductivity*, 198(3), 261-272.

[10]. Yusmi, H., Kechik, M. M. A., & Abd-Shukor, R. (2016). Effect of Yb2O3 nanoparticle addition on superconducting properties of BSCCO (2223)/Ag tapes by acetate precipitation method. *Pertanika Journal of Science and Technology*, 2(1), 1-9.