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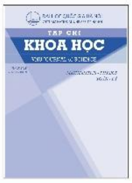

Original Article

Excess Conductivity Analyses in Bi-Pb-Sr-Ca-Cu-O Systems Sintered at Different Temperatures

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Abstract: This paper studies the effects of the sintering temperature on crystal structure, critical temperature (T_c) and excess conductivity of Bi-Pb-Sr-Ca-Cu-O (BPSCCO) system. Bulk BPSCCO samples were fabricated by the solid-state reaction method. Four different temperatures of 835 °C, 840 °C, 845 °C, and 850 °C were applied to sinter four different samples. The crystal structure of the samples was investigated through X-ray diffraction measurements (XRD) and scanning electron microscopy (SEM). Superconductivity of the samples was analyzed by the temperature-dependent resistivity measurement. The experimental results showed the existence of both Bi-2223 and Bi-2212 phases in all samples. Quantitatively, the volume fraction of the Bi-2223 phase was found to increase from 53.56% to 75.97%, and the average grain size of Bi-2223 phase was observed to enlarge from 57.95 nm to 86.50 nm as the sintering temperature increased from 835 °C to 850 °C. In addition, the excess conductivity analyses based on the theory of Aslamazov - Larkin (AL) and Lawrence - Doniach (LD) showed decreases in the coherence lengths ($\xi_c(0)$) from 1.957 Å to 1.565 Å and the effective inter-layering spacing (d) from 79.7 Å to 64.5 Å. Meanwhile, the interlayer coupling strength (J) between two CuO_2 planes was estimated to increase from 0.00083 to 0.00137. These results might be evidence to conclude that the increasing of the sintering temperature obviously improves the superconductivity in the BPSCCO system.

Keywords: BPSCCO, Bi-2223, Bi-2212, T_c, excess conductivity

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1. Introduction

Since the discovery of high temperature superconductors $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4+\delta}$ (BSCCO), many studies have been carried out on this system with the aim to enhance their superconducting properties [1, 2]. Depending on the number of CuO_2 layers (n) in a unit cell of BSCCO, this system has three different superconducting phases [3, 4]. Those phases are Bi-2201 (n = 1), Bi-2212 (n = 2) and Bi-2223 (n = 3) with T_c = 33 K, 80 K, 110 K, respectively [5, 6]. Although the Bi-2223 phase has been revealed to be the most important phase of BSCCO for power related applications, the fabrication of this phase has been reported to be relatively difficult. Fabrication conditions of the Bi-2223 phase required sintering at specified temperature and time. The previous studies pointed out that partial substitutions of Pb at the Bi site in the crystal structure might help to reduce the sintering temperature and time. The appearance of Ca_2PbO_4 was believed to be a reason for enhancing the formation and stabilization of Bi-2223 phase [7-9]. Many studies showed that the optimal content ratio of Bi:Pb for the formation of Bi-2223 phase was 1.6:0.4 [10].

The formation of conducting pairs just above T_c has been attributed to superconducting order parameter fluctuations, which plays an important role in specifying the superconducting properties [11-14]. For better understanding the intrinsic properties of the superconductors such as coherence lengths, the effective inter-layering spacing and the interlayer coupling strength between the two CuO_2 layers, variations of excess conductivity versus reduced temperature ($t = (T - T_c)/T_c$) were analyzed by using the Aslamazov-Larkin (AL) and Lawrence - Doniach (LD) models [11, 13].

By varying the sample preparation conditions, the formation of the Bi-2223 phase in the BPSCCO samples would be accelerated. Recently, Kocabas et al. studied the effect of sintering temperature on Sb substituted BPSCCO superconductor [15]. The results showed the variations of macroscopic properties such as volume fractions of superconducting phases, surface morphology and T_c with respect to the sintering temperature. Although the effects of sintering temperature on the microscopic properties of the BPSCCO samples were obtained from AL and LD models, they were not widely reported.

In this work, we will apply these two models to investigate the excess conductivity as well as the dependence of superconductivity on the sintering temperatures of the four pure $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ superconductors sintered at four different temperatures. These temperatures were chosen as sintering temperature due to the fact that the melting point of the system is 855 °C and the formation of Bi-2212 phase is 820 °C [8]. The optimal sintering temperature for the fabrication of the BPSCCO sample with a high-volume fraction of the Bi-2223 phase will be investigated.

2. Experiment

Solid-state reaction technique has been reported to be one of the most effective methods to fabricate polycrystalline superconductors [16]. The superconducting samples used in this work with the formula $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ were prepared from the precursors: Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$ and CuO with purities of 99.9%. Based on the stoichiometric ratio of the samples, the amounts of the starting materials were weighed, mixed and ground. The thoroughly ground mixture was pressed into pellets and subjected to a four-stage calcination process in air. Each stage lasted for 48 hours at 670 °C, 750 °C, 800 °C and 820 °C. Between two consecutive stages, the grinding - pressing steps were re-applied. To compare the formation of the Bi-2223 phase, four different $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ samples were sintered for 168 hours at different temperatures of 835 °C, 840 °C, 845 °C and 850 °C, respectively. Finally, all the samples were freely cooled to the room temperature. The fabricated samples were then named as M0_835, M0_840, M0_845 and M0_850.

Crystalline structure of the samples was investigated using X-ray diffraction technique (XRD, Miniflex 600) using Cu-K radiation. The surface morphology of the samples was examined by using the scanning electron microscopy (SEM, Nova Nano SEM 450). The superconducting properties of the samples were investigated via the temperature dependence of electrical resistivity measurement performed by using the four-probe method in the temperature range from 80 - 300 K (closed cycle helium).

3. Results and Discussion

3.1. Crystallinity of the Samples

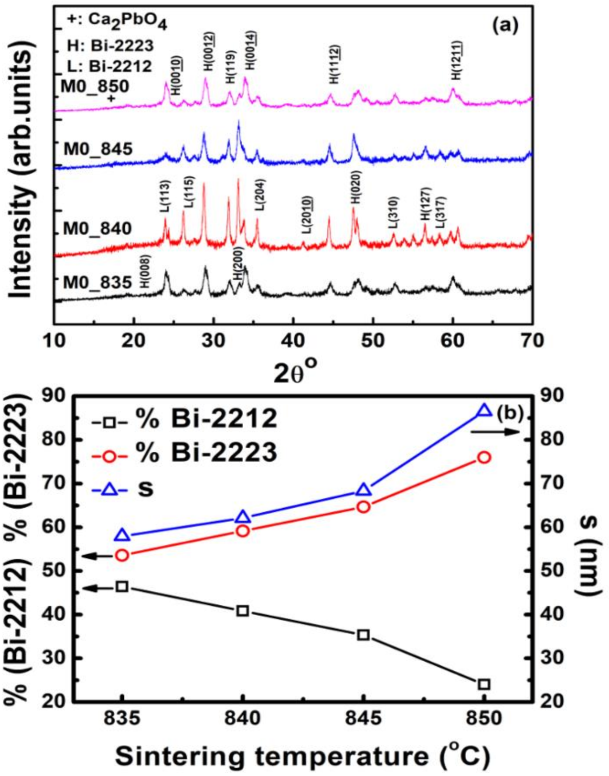

Figure 1. (a) X-ray diffraction diagram and (b) the volume fraction for Bi-2212 phase and Bi-2223 phase of the BPSCCO samples sintered at temperatures of 835 °C, 840 °C, 845 °C, and 850 °C.

Figure 1a shows the XRD patterns of all samples. The 2θ diffraction angle was set from 10° to 70°. It is observed that the four samples consisted of two superconducting phases: Bi-2212 (marked as "L") and Bi-2223 (marked as "H"). In addition, the presence of secondary Ca_2PbO_4 phase (marked as "+") has been attributed to the result of partial reaction between Pb with other precursors during sample fabrication [7-10]. The Ca_2PbO_4 phase plays an important role in accelerating the formation of Bi-2223 phase [7, 10].

In order to investigate the change in the formation of superconducting phases at the four sintering temperatures, the volume fraction of superconducting phases (%Bi-2223 and %Bi-2212) were calculated by using the formula [17]:

$$\%\mathrm{Bi}-2223=\frac{\sum \mathrm{I}_{2223}}{\sum \mathrm{I}_{2223}+\sum \mathrm{I}_{2212}}\times 100\%$$

$$\%\mathrm{Bi}-2212=\frac{\sum \mathrm{I}_{2212}}{\sum \mathrm{I}_{2223}+\sum \mathrm{I}_{2212}}\times 100\%$$

Another feature used to analyze the formation of the superconducting phases is the average grain size (s). The value of s has been also estimated from the XRD results, according to Sherrer's formula [18]:

$$\mathrm{s}=\frac{0.941\lambda}{\mathrm{B}\cos\theta_{\mathrm{B}}}$$

where λ was the wavelength of Cu Kα X-ray radiation, θ_{B} was the Bragg diffraction angle and B was the full width at half maximum of the selected diffraction peak. Variations of the estimated data of %Bi-2223, %Bi-2212 and s versus sintering temperature are presented in Figure 1b.

It is seen in Figure 1b that the formation of Bi-2223 phase was clearly accelerated as the sintering temperature increased. Specifically, the %Bi-2223 increased gradually from 53.56% for M0_835 sample to 75.97% for M0_850 sample. Similarly, the s was also observed to have increased from ~ 57.95 nm for M0_835 sample to ~ 86.5 nm for M0_850 sample.

3.2. Surface Morphology of Samples by Scanning Electron Microscopy (SEM)

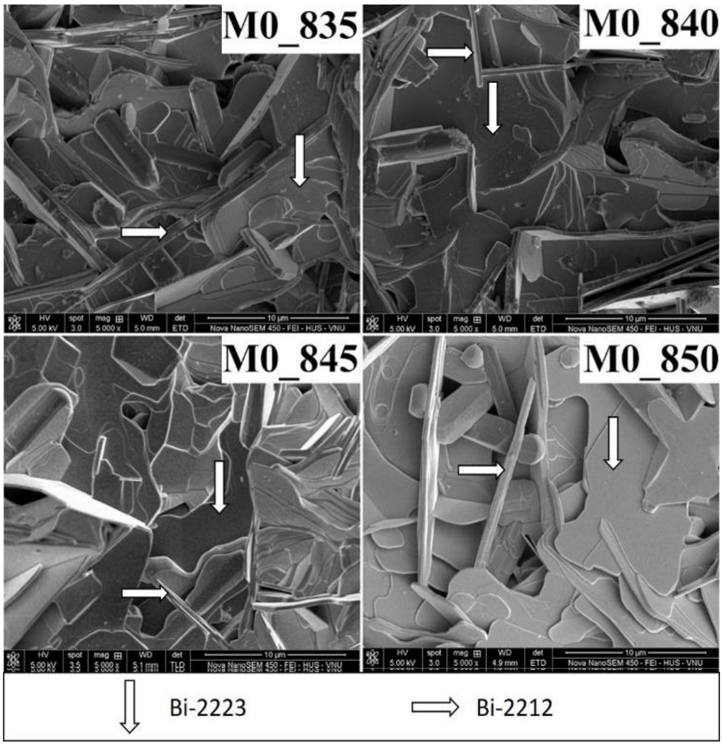

Figure 2. SEM pictures of the BPSCCO samples sintered at temperatures of 835 °C, 840 °C, 845 °C, and 850 °C.

Surface morphologies of the samples were investigated through the SEM images, those are given in Figure 2. The SEM images of the samples showed that all samples consisted of Bi-2223 and Bi-2212 phases which were found in the forms of plate-like and needle-like grains, respectively [19]. In the literature, the Bi-2223 grains are likely to grow in the a-b plane direction, implying that the plate-like grains were nearly c-axis oriented. We can clearly see from the XRD result, most of the H peaks were c-axis oriented. It was clearly observed that the Bi-2223 grains were likely to enlarge while the grain boundary decreased as the sintering temperature increased. The trend was similar to the results of the X-ray analysis. The small spherical grains were identified to be the Ca_2PbO_4 phase [19]. Hence, it might be said that the formation of the Bi-2223 phase was accelerated when Ca_2PbO_4 existed in samples. These observations were found to be in agreement with the results obtained from the X-ray analyses. These results were completely consistent with other studies [5, 20, 21]. From these results, it might be said that the higher sintering temperature, the more Bi-2223 phases were created. Experimentally, when the sintering temperature reached 855 °C, the sample started to melt, so the sintering temperature was intercepted at 850 °C.

3.3. Superconductivity of the Samples

In order to examine the superconductivity of the samples, temperature dependences of resistivity of the samples were measured. The experimental results are given in Figure 3. For all samples, the metallic-behavior existed at the high temperature region, where the resistivity of all samples linearly decreased versus temperature. As temperature continuously decreased, the superconducting transition occurred. The transition width (ΔT_c) was determined by the formula $\Delta T_c = T_{c/on} - T_{c/off}$. Definitions of $T_{c/on}$ and $T_{c/off}$ were explained as follows: $T_{c/on}$ was the temperature at which the resistivity of the sample deviated from the linearity; and $T_{c/off}$ was the temperature at which the resistivity of the sample was completely reduced to zero. In other words, the two temperatures corresponded to the partial and completed transitions of superconducting phases [22].

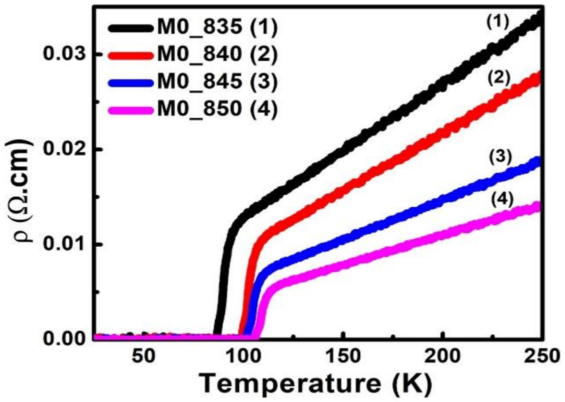

Figure 3. Temperature dependence of the resistivity of the BPSCCO samples sintered at temperatures of 835 °C, 840 °C, 845 °C, and 850 °C.

In order to find the mean field transition temperature (T_c) of the samples, a graph of the first derivative of resistivity versus temperature (dR / dT) is exhibited in Figure 4. The value of T_c was defined as the position of the peak in dR / dT curve. Values for $T_{c/on}$, $T_{c/off}$, T_c and ΔT_c are listed in Table 1.

The hole concentration on the CuO_2 plane is determined by the following formula [21]:

$$p = 0.16 - [(1 - \frac{T_{c/off}}{T_{c/max}})/82.6]^{1/2}$$

Table 1. Phase transition temperatures ($T_{c/on}$, $T_{c/off}$, $T_{c)}$, transition width (ΔT_c), hole concentration (p) and the parameters deduced by analyzing excess conductivity of BPSCCO samples sintered at temperatures of 835 °C, 840 °C, 845 °C, and 850 °C

Parameters	M0_835	M0_840	M0_845	M0_850
$T_{c/on}$ (K)	97.75	107.77	111.55	115.78
$T_{c/off}$ (K)	87.24	98.03	102.48	106.95
T_c (K)	90.02	102.52	104.74	109.38
ΔT_c	10.51	9.74	9.07	8.83
p	0.11	0.123	0.131	0.141
λ_{CR}	0.317	0.316	0.323	0.321
λ_{3D}	0.533	0.497	0.501	0.515
λ_{2D}	1.037	1.0345	1.050	1.068
T_{LD} (K)	91.377	103.834	106.163	110.872
10J	0.0083	0.0128	0.0136	0.0137
β (K^{-1})	0.068	0.055	0.049	0.043
d (Å)	79.7	75.9	69.7	64.5
$\xi_c(0)$ (Å)	1.957	1.791	1.649	1.565

Through the data from Table 1, it is clearly seen that increasing the sintering temperature directly affects T_c and p. The T_c of the sample gradually increases from 90.02 K for M0_835 sample to 109.38 K for M0_850 sample. These results are in good agreement with the T_c of the samples given in previous studies [5, 7, 8, 20]. It has been widely reported in the literature that T_c of Bi-2212 phase is 80 K and that of Bi-2223 phase is 110 K. The obtained values of T_c might reveal the co-existence of the two superconducting phases Bi-2212 and Bi-2223 in our samples. From the calculation of hole concentration, it is found that the higher the sintering temperature is, the greater the hole concentration and the more apparent the superconductivity are.

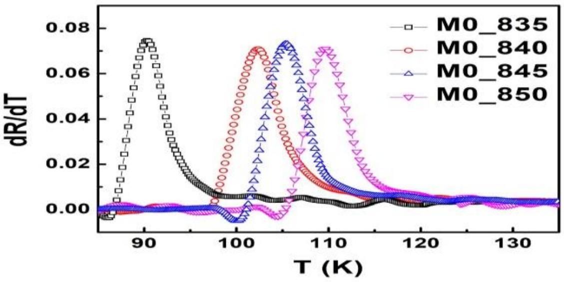

Figure 4. First derivative of resistivity against temperature (dR/dT) of the BPSCCO samples sintered at temperatures of 835 °C, 840 °C, 845 °C, and 850 °C.

3.4. Excess Conductivity

The results of the above measurements have revealed the macroscopic properties of the BPSCCO superconducting system. In order to further investigate microscopic properties such as coherence length $\xi_c(0)$, the effective inter-layering spacing (d), the interlayer coupling strength (J) between two CuO_2 planes and the behavior of conducting pair at critical temperature, the theoretical explanation of excess conductivity ($\Delta\sigma$) provided by Aslamazov and Larkin (AL) using a microscopic approach in the mean field region (MFR) has been used [23]. $\Delta\sigma$ is given by:

$$\Delta\sigma = At^{-\lambda} \ (1)$$

Where $t = (T - T_c)/T_c$ is the reduced temperature, λ is the Gaussian critical exponent related to the conduction dimensionality expressed as follows: $\lambda = 0.3$ for critical fluctuations (CR), $\lambda = 0.5$ for 3D fluctuations, $\lambda = 1.0$ for 2D fluctuations and $\lambda = 3.0$ for short wave fluctuations (SWF). A is a temperature independent constant given by equation: $A = \frac{e^2}{32\hbar\xi_c(0)}$ for 3D fluctuations (2) and $A = \frac{e^2}{16\hbar d}$ for 2D fluctuations (3). In practice, the excess conductivity is obtained from the equation [12]:

$$\Delta\sigma = \sigma(T) - \sigma_n(T) \ (4)$$

where $\sigma(T) = 1/\rho(T)$ is the experimental conductivity, $\sigma_n(T) = 1/\rho_n(T)$ is the extrapolated conductivity from the metallic-behavior region.

AL theory was extended by Lawrence and Doniach (LD) for strong anisotropic superconductor [11, 24]. According to this theory, the interlayer coupling strength between two layers of CuO_2 (J) is given by: $J = (\frac{2\xi_c(0)}{d})^2$ (5). Then, excess conductivity is given by:

$$\Delta\sigma = \frac{e^2}{16\hbar d}(1 + J/t)^{-1/2}t^{-1} \ (6)$$

The crossover temperature between 2D and 3D fluctuations was named as T_{LD} and expressed by: $T_{LD} = T_c(1 + J)$ (7). For the weak coupling, the excess conductivity equation became: $\Delta\sigma = At^{-1}$ (8).

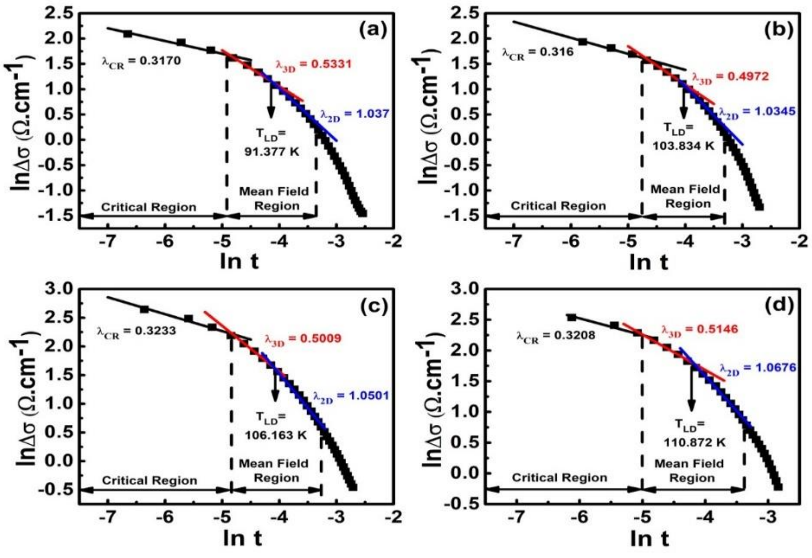

Figure 5. Double logarithmic plot of excess conductivity as a function of reduced temperature t for (a) M0_835, (b) M0_840, (c) M0_845, and (d) M0_850. The black lines are the fitting line of CR in the LD models. The red and black fitting lines respectively correspond to the 3D and 2D regions in the MFR.

The double-logarithmic plots of $\Delta\sigma$ versus t of the samples are presented in Figure 5. From the Gaussian critical exponent obtained by fitting the AL model in our data, different regimes were separated: the mean field regime (MFR) and the critical regime (CR). The values of T_{LD} were obtained from the intersection of 2D and 3D fitting lines. The temperature just above T_{LD} in MFR is the 2D fluctuation which has critical exponent around $\lambda = 1$. This is the regime where the conducting pairs start to form and its movement was limited in CuO_2 layers [13, 25]. When the temperature further decreases to lower than T_{LD}, the CuO_2 layers are connected by Josephson channeling that allows the conducting pairs to move between two CuO_2 layers [26, 27]. This region is 3D fluctuation with the critical exponent around $\lambda = 0.5$. The critical regime with critical exponent $x = 0.3$ is where samples fully change to superconductors. As can be seen from Table 1, the three critical exponents of 2D fluctuation, 3D fluctuation and CR are ~1, 0.51, 0.32, respectively. These results are reasonable according to the AL theory.

As the sintering temperature increased, T_{LD} and interlayer coupling strength J also increased showing that a large quantity of conducting pair formed [25-28]. The slope β of $1/\Delta\sigma$ versus temperature is expressed as [26]: $\beta = \frac{16\hbar d}{e^2 T_{ext}}$ (9), where T_{ext} is extrapolated temperature obtained from the intersection of the extrapolation of the plot with the T axis as shown in inset of Figure 6. By using slope β and equation (1) - (5) - (6) - (8), the value of d and $\xi_c(0)$ have been determined. It is found that higher sintering temperatures induce the decreases in both d and $\xi_c(0)$, which leads to the improvement in superconductivity in the BPSCCO samples [29]. The sintering temperatures might affect the critical current density of our samples since the surface morphologies of the samples were observed to change. However, we did not measure this property yet; therefore, further investigations will be reported later.

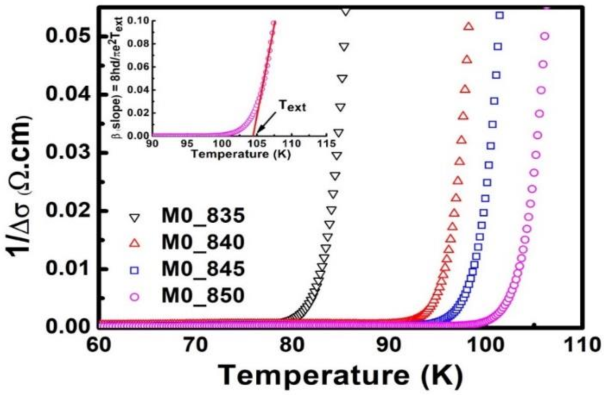

Figure 6. Temperature dependence of $\Delta\sigma^{-1}$ of the BPSCCO samples sintered at temperatures of 835 °C, 840 °C, 845 °C, and 850 °C. β can be determined from the linear fitting lines as shown in the inset.

4. Conclusion

The BPSCCO high-temperature superconductors have been successfully fabricated at different sintering temperatures of 835 °C, 840 °C, 845 °C and 850 °C. The formations of the two superconducting phases of Bi-2223 and Bi-2212 were clearly observed. With increasing sintering temperature, volume fraction of the Bi-2212 phase decreased, and that of the Bi-2223 phase increased. Superconductivity of the samples is significantly improved with increasing sintering temperature: the values of critical temperature T_c, crossover temperature T_{LD}, interlayer coupling strength J were found to increase while the transition width ΔT_c, coherence length $\xi_c(0)$ and the effective layer thickness d decreased. It might be concluded that the temperature of 850 ^oC was the optimal sintering temperature for the fabrication of the BPSCCO sample with a high-volume fraction of the Bi-2223 phase.

Acknowledgments

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