ORIGINAL PAPER

The Effect of CdO Nanoparticles on the Structure and Magnetic Properties of Bi$_{1.64}$Pb$_{0.36}$Sr$_2$Ca$_{2-x}$Cd$_x$Cu$_3$O$_y$ Superconductors

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Abstract In this paper, the CdO nanoparticles were synthesized by sol-gel method Then, the effects of CdO nanoparticles on the structure and weak-link behavior of the Bi–Pb–Sr–Ca–Cu–O system, which has a nominal starting composition of Bi$_{1.64}$Pb$_{0.36}$Sr$_2$Ca$_{2-x}$Cd$_x$Cu$_3$O$_y$ (BPSCCC) ($x = 0.0, 0.01, 0.02, 0.03, 0.04, 0.1, 0.5, 1$, and 2), were studied. The BPSCCC superconductor was made using a solid state reaction method. The structural analysis of BPSCCC was carried out using X-ray diffraction (XRD) measurements. The standard four-probe method was carried out to measure critical current density. The temperature dependence of ac magnetic susceptibility of samples was measured as a function of ac magnetic field amplitude by using a Lake Shore ac susceptometer. The results of XRD and ac magnetic susceptibility indicated that the Bi-2201 and Bi-2212 phases reduce the critical current density and critical temperature. In addition, the small amount of doping ($x = 0.01$) was found to improve the superconducting properties of the Bi–Pb–Sr–Ca–Cu–O system.

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1 Introduction

The Bi$_2$Sr$_2$Ca$_{n-1}$Cu$_n$O$_{4+2n}$ superconductor consists of three different phases which are called the Bi-2201 phase ($n = 1$ $T_C \approx 20$ K), Bi-2212 phase ($n = 2$, $T_C \approx 85$ K), and Bi-2223 phase ($n = 3$, $T_C \approx 110$ K). Here, we have used the abbreviation Bi-2201, Bi-2212, and Bi-2223 phases for $n = 1, 2$, and 3, respectively. In these phases, the Bi-2223 is the most attractive, because it has the highest superconducting transition temperature, T_C, about 110 K [1]. It is generally known that chemical doping plays a signification role in high-T_C superconductivity. However, the pure phase of Bi-2223 is difficult to obtain, and its formation is influenced by many preparation conditions such as composition; annealing time; temperature, atmosphere, and pressure during sintering; type and quantity of the dopant; heat treatment method; and operational procedures [2–4]. Although a large number of elements such as Pb [5], Gd [6], Sb [7], Ag [8], Cd [9, 10], and Zr, Hf, Mo, and W [11] have been doped into the Bi–Pb–Sr–Ca–Cu–O system, it seems that the doping CdO nanoparticles have not been properly studied. The nanoparticles of materials have attracted great interest in recent years because of their unique chemical and physical properties, which are different from their bulk properties. There have been some efforts to synthesize nanoparticles of CdO by different methods, such as mechanochemical reaction and chemical methods. In this paper, the CdO nanoparticles were obtained using a chemical method [12–14]. The ac magnetic susceptibility is widely used for the determination and characterization of the intergranular component in the high-T_C superconductors. The dependence of ac magnetic susceptibility on the ac field amplitude (H_{ac}) was determined using a pining strength parameter. However, the major limitations of the Bi-based

superconductor are the intergrain weak links and the presence of additional phases. Investigations reveal that the Bi-2201 phase is located mainly between superconducting grains, preventing the supercurrent flow [15–17]. A tremendous effort has been applied to improve the links between the grains, and for this propose, the doping of various elements has been done. Whereas the ionic radius of Cd^{+2} is close to Ca^{+2}, in this research, we have systematically and rigorously studied the effects of CdO nanoparticle substitution for Ca in $Bi_{1.64}$ $Pb_{0.36}$ Sr_2 Ca_{2-x} Cd_x Cu_3O_y ($x = 0.0, 0.01, 0.02, 0.04, 0.1, 0.5,$ 1, and 2) superconductor. The critical current density, critical temperature, magnetic susceptibility, and structure properties of a series of samples were also taken into consideration.

2 Material and Methods

Samples of $Bi_{1.64}Pb_{0.36}Sr_2Ca_{2-x}Cd_xCu_3O_y$ with x = 0.0, 0.02, 0.04, 0.1, 0.5, 1, and 2 were prepared using a solid state reaction method. The Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$, and CuO powders with high purity and CdO nanoparticles were used. The required quantities of reagents were weighed ($\Delta m = 10^{-4}$ mg) and mixed. In order to prevent the growth of additional phases during the process, the powders were ground and milled for an hour. The mixed powders were calcined in air at 820 °C for 15 h. The materials were reground and pressed into the bar shape samples with dimensions of $2.4 \times 5.8 \times 20.6$ mm^3 under the pressure of 820 MPa. The samples were put in the furnace, and the procedure of sintering was done in an air atmosphere. Some of the samples were given the Meissner effect test in liquid nitrogen. The critical current densities and critical temperature of the samples were measured using the standard four-probe method. The ac magnetic susceptibility measurements were performed using a Lake Shore ac susceptometer, model 7000. The X-ray diffraction (XRD) patterns of the samples were taken with a Philips X-ray diffractometer model PW1840.

3 Results and Discussion

Figure 1a, b shows the results of XRD and SEM of CdO nanoparticles. It is clear from Fig. 1a that the CdO nanoparticles are single phase and cubic structure with a lattice parameter of $a = 4.7$ Å without showing any detectable impurities. The observed diffraction peaks were found to correspond to those of the standard pattern (JCPDS # 05-0640). Also, Fig. 1b shows that the average particle size is approximately 90 nm.

The XRD measurements of the samples are shown in Fig. 2. Based on our XRD measurements, we observed that by substitution of CdO nanoparticles for Ca at an amount of $x = 0.01$, the Bi-2223 phase increases in the sample and CdO nanoparticles seem to act as a phase stabilizer. As one can see, the samples consist of a mixture of Bi-2223, Bi-2212, Bi-2201, and $Bi_2Sr_{1.5}Ca_{0.5}Cu_2O_{8.4}$ phases as the major constituents. From the XRD measurements, it was revealed that the volume of Bi-2223 phase decreases with an increasing CdO nanoparticle content up to $x = 0.1$ (sample f). The XRD results show that the optimum amount of CdO nanoparticles is about $x = 0.01$ (sample b), which characterizes the highest volume fraction of Bi-2223 phase. The volume fraction of the phases can be estimated by various methods [18]. We have used all the peaks of the Bi-2223, Bi-2212, Bi-2201, and $Bi_2Sr_{1.5}Ca_{0.5}Cu_2O_{8.4}$ phases for the estimation of the volume fractions of the phases, and we ignored the voids. The volume fractions of the phases for all the samples with annealing times of 90, 180, and 270 h were given in Tables 1, 2, and 3, respectively. Also, it can be seen from Table 3 that the maximum volume fraction of Bi-2223 phase is obtained for the sample of $x = 0.01$. By increasing the amounts of CdO nanoparticles to more than 0.01, the volume fraction of the Bi-2223 phase decreases.

The ρ–T curves of the samples at an annealing time of 270 h with different contents of CdO nanoparticles are shown in Fig. 3. From these curves, one can see that by increasing the content of CdO nanoparticles, the critical temperature of the samples will be decreased. So, the optimum value of CdO nanoparticles is 0.01.

Figure 4 shows the temperature dependence of the real, χ' and imaginary, χ'' parts of ac magnetic susceptibility for samples with amounts of $x = 0.0$ and 0.01 in an ac field of 5 A/m with a frequency of 333 Hz which is applied parallel to the long dimension of samples. In particular, the imaginary component, χ'' of the ac magnetic susceptibility is widely used to probe the nature of weak links in polycrystalline superconductors. It is also employed to estimate some of the important physical properties, such as the critical current density and effective volume fraction of the superconducting grains. The real part of the ac magnetic susceptibility, χ' in polycrystalline samples shows two drops as the temperature is lowered below the onset of the diamagnetic transition, and correspondingly, the derivative of the $\chi'(T)$ displays two peaks. The first sharp drop at the critical temperature is due to the transition within the grains, and the second gradual change is due to the occurrence of the superconducting coupling between grains. The imaginary part χ'' shows a peak which is a measure of the dissipation in the sample. When the peak of χ'' shifts to lower temperatures and broadens the intergranular coupling between the grains, the critical current density decrease [19,

Fig. 1 **a** XRD pattern and **b** SEM image of CdO nanoparticles
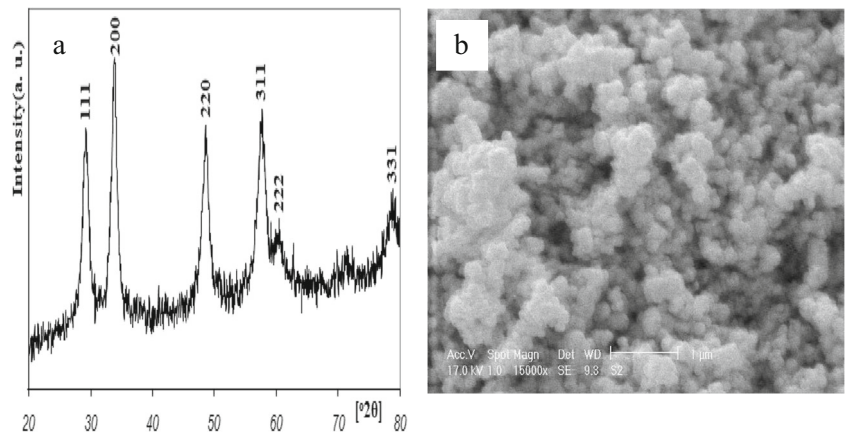
20]. It can be seen from the curves in Fig. 4 that the intergranular coupling between the grains and the critical current density of the sample of $x = 0.01$ is better than that of the sample of $x = 0.0$.

Figure 5 shows the results of ac magnetic susceptibility measurements for the sample of $x = 0.01$ and an annealing time of 270 h for various ac fields with a frequency of 333 Hz. The losses in the type II superconductors arise from the viscous flux flow motion and hysterics due to the pinning of flux vortices [21]. Thus, pinning losses are independent of frequency, but dependent on the amplitude of field, whereas flux flow losses depend on the frequency not the amplitude of field. Many researchers have observed that the complex ac magnetic susceptibility of high-T_C superconductors is weakly dependent on the frequency and

strongly on the amplitude of ac magnetic field. It has been shown that the dependence of ac magnetic susceptibility on ac magnetic field amplitude H_{ac} was determined using a pinning strength parameter. Diamagnetic onset temperature of the intrinsic superconducting transition for these samples is about 107 K. It is clear from Fig. 5 that, as the magnetic field increases, the peak of χ'' shifts to lower temperatures and broadens. Also, the effect of the magnetic field on the intergranular component in the real part (χ') shifts to lower temperatures. The amount of this shift is a function of the magnetic field amplitude, H_{ac}, and it is also proportional to the strength of the pinning force. When the pinning force becomes smaller, it depresses the critical current [22, 23].

Figure 6 reveals the results of the ac magnetic susceptibility measurements for the samples of $x = 0.01$, 0.04, and 0.1 with and an annealing time of 270 h for an ac magnetic field of 5 A/m with a frequency of 333 Hz. From Fig. 6, it is clear that as the amounts of CdO nanoparticles increase, the peak of χ'' shifts to lower temperature and broadens. The peak shift of the sample of $x = 0.1$ is much more than that of the others. It can be seen from the curves that the intergranular coupling and critical current density of the sample of $x = 0.01$ are better than those of other samples. When x past 0.01, the superconductivity properties such as the magnetic susceptibility, critical temperature, and critical current density were decreased significantly (see sample of $x = 0.1$) [24, 25].

Figure 7 shows the results of the ac magnetic susceptibility measurements of the sample of $x = 0.01$ with annealing times of 90, 180, and 270 h for an ac magnetic field of 5 A/m with a frequency of 333 Hz. From Fig. 7, it can be seen that by increasing the annealing time up to 270 h, the peak of χ'' shifts to higher temperature. These results indicated that the intergranular coupling between grains increases by increasing the annealing time up to 270 h.
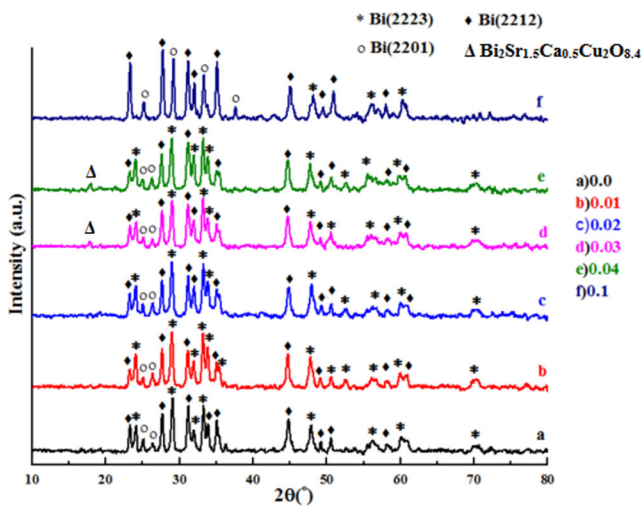
Fig. 2 XRD patterns of samples with different CdO nanoparticle contents: *a* 0.0, *b* 0.01, *c* 0.02, *d* 0.03, *e* 0.04, and *f* 0.1 and an annealing time of 270 h

**Table 1** Relative volume fractions of Bi-2223, Bi-2212, and Bi-2201 with an annealing time of 90 h

| x	0.0	0.01	0.02	0.03	0.04	0.1
Bi-2223	33.42	50.20	47.74	42.30	56.89	5.83
Bi-2213	61.63	42.78	46.02	51.70	36.15	62.94
Bi-2201	4.95	7.02	6.24	6.00	6.96	31.23

Table 2 Relative volume fractions of Bi-2223, Bi-2212, and Bi-2201 with an annealing time of 180 h

x	0.0	0.01	0.02	0.03	0.04	0.1
Bi-2223	39.29	52.64	57.90	47.81	56.17	7.79
Bi-2213	54.50	39.25	36.21	45.06	35.98	69.98
Bi-2201	6.21	8.11	5.89	7.13	7.85	22.23

Table 3 Relative volume fractions of Bi-2223, Bi-2212, and Bi-2201 with an annealing time of 180 h
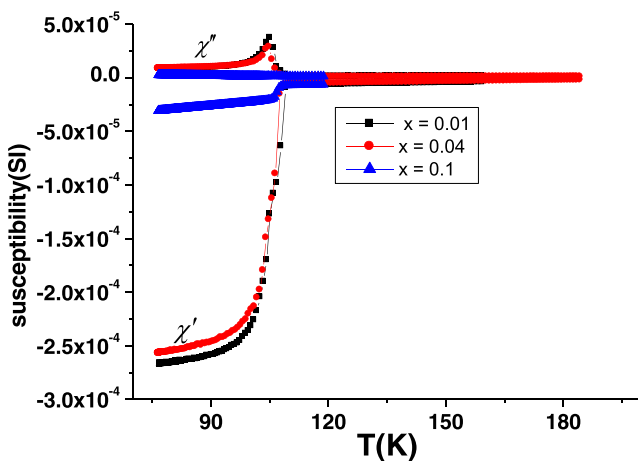
x	0.0	0.01	0.02	0.03	0.04	0.1
Bi-2223	40.21	63.51	53.24	48.03	56.18	8.02
Bi-2213	54.67	33.48	40.44	43.83	33.10	62.53
Bi-2201	5.12	4.01	6.32	6.01	6.19	29.45
$Bi_2Sr_{1.5}Ca_{0.5}Cu_2O_{8.4}$	–	–	–	3.23	4.53	–
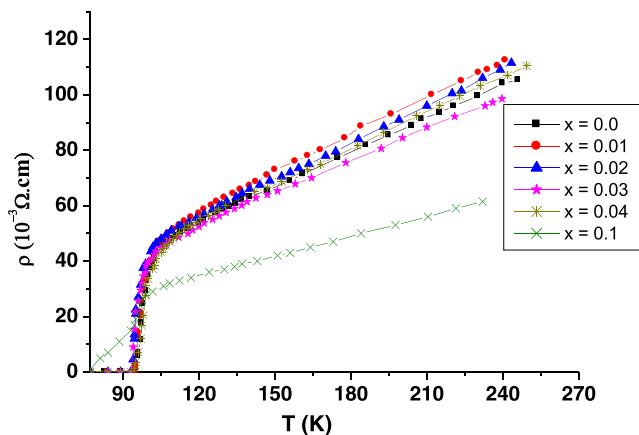
Fig. 3 The ρ–T curves for different CdO nanoparticle contents: 0.0 (*black square*), 0.01 (*red circle*), 0.02 (*blue triangle*), 0.03 (*pink star*), 0.04 (*asterisk*), and 0.1 (*multiplication sign*) with an annealing time 270 h
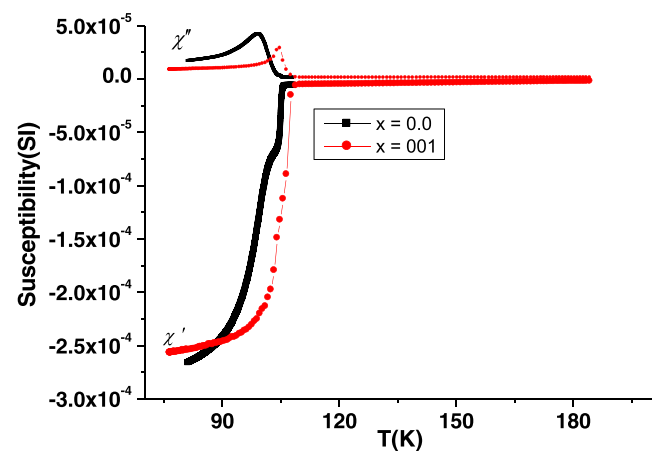
Fig. 4 Temperature dependence of the magnetic susceptibility for samples with CdO nanoparticle contents: 0.0 (*black square*) and 0.01 (*red circle*) and an annealing time of 270 h with a field of 5 A/m
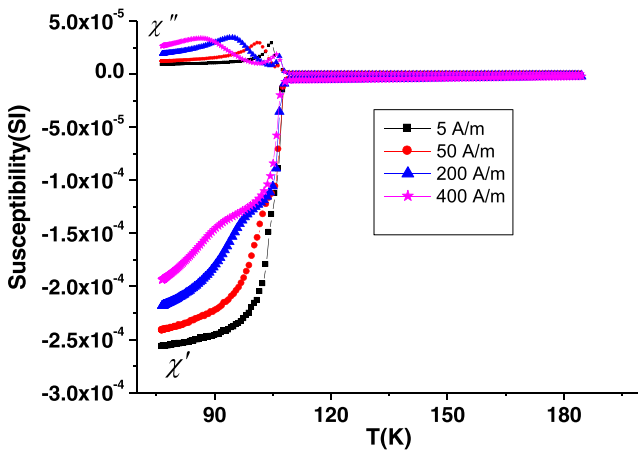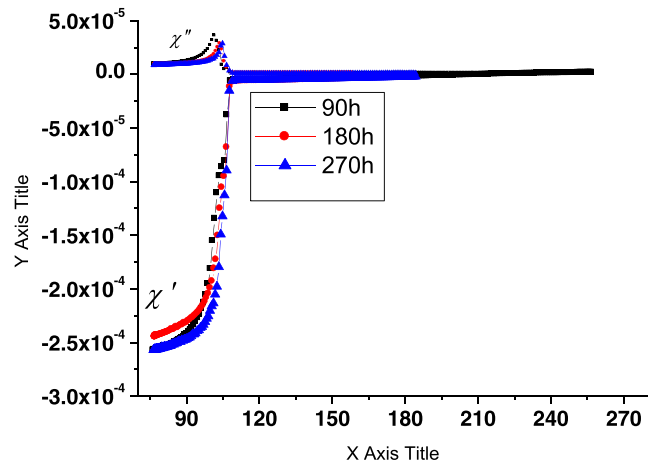
Fig. 5 Temperature dependence of the magnetic susceptibility with fields 5 A/m (*black square*), 50 A/m (*red circle*), 200 A/m (*blue triangle*), and 400 A/m (*pink star*) for sample with a CdO nanoparticle content of $x = 0.01$ and an annealing time of 270 h

Fig. 7 Temperature dependence of the magnetic susceptibility with annealing times: 90 h (*black square*), 180 h (*red circle*), and 270 h (*blue triangle*) for sample with a CdO nanoparticle content of $x = 0.01$ and a field of 5 A/m

In Fig. 8, we compare our result from that of Mousavi et al. [26] which the CdO bulk has been doped in the same sample with this research. It shows the results of the ac magnetic susceptibility measurements of the samples with a CdO bulk amount of 0.04 and a CdO nanoparticle amount of 0.01 with an annealing time of 270 h for an ac magnetic field of 5 A/m with a frequency of 333 Hz. From Fig. 8, it can be seen that the critical current density, intergranular coupling between grains, and volume fraction of Bi-2223 phase in a CdO nanoparticle amount of $x = 0.01$ are more than those of the sample with a CdO bulk amount of $x = 0.04$. Study of superconductivity properties of CdO nanoparticles
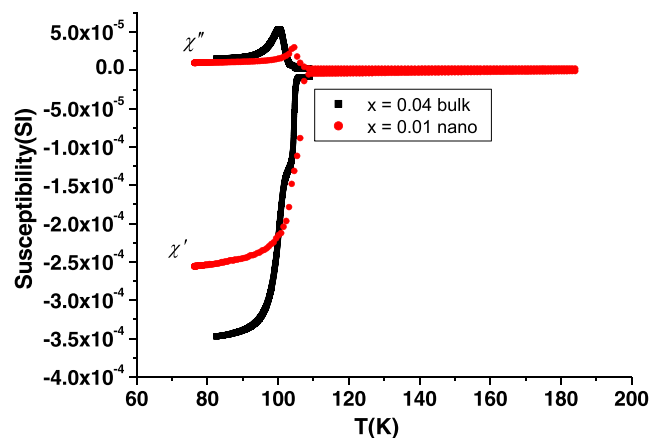
on $Bi_{1.64}Pb_{0.36}Sr_2Ca_{2-x}Cd_xCu_3O_y$ superconductors indicated that the optimum amount of the sample is $x = 0.01$, whereas in the bulk state, it is $x = 0.04$. This is due to the fact that the nanoparticles have more capability to spread in the sample with respect to their bulk. Thus, the less percent of nanoparticles will improve the superconducting properties [27–29].

Figure 9 shows the $V-J$ curves, taken at liquid nitrogen temperature, for the samples of $x = 0.0, 0.01, 0.02, 0.03$, and 0.04 at an annealing time of 270 h. One can observe that by increasing the amounts of CdO nanoparticles, the critical current densities of the samples increase. Our observations suggest that not only CdO nanoparticles do act as a Bi-2223

Fig. 6 Temperature dependence of the magnetic susceptibility for samples with CdO nanoparticle contents: 0.01 (*black square*), 0.04 (*red circle*), and 0.1 (*blue triangle*) and an annealing time of 270 h with a field of 5 A/m

Fig. 8 Temperature dependence of the magnetic susceptibility for samples with CdO bulk amount of 0.04 (*black square*) and a CdO nanoparticle amount of 0.01 (*red circle*) and an annealing time of 270 h with a field of 5 A/m
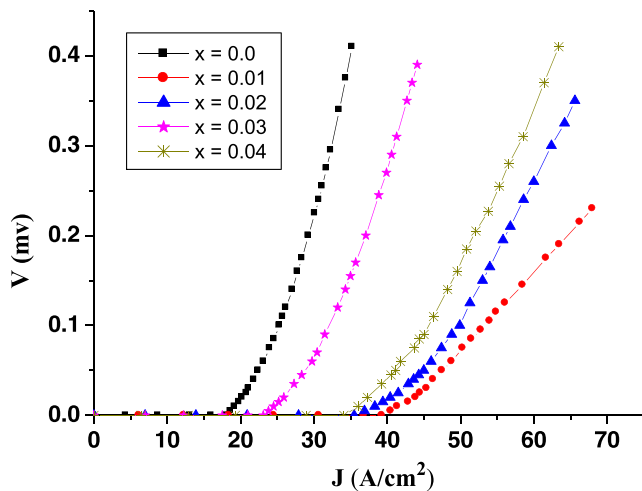
Fig. 9 The $V-J$ curves for different CdO nanoparticle contents: 0.0 (*black square*), 0.01 (*red circle*), 0.02 (*blue triangle*), 0.03 (*pink star*), and 0.04 (*asterisk*) and an annealing time of 270 h

the sample of $x = 0.01$ are greater than those of the others. The XRD results reveal that the sample of $x = 0.01$ with an annealing time of 270 h has the highest percentage of Bi-2223 phase. Also, our results show that the maximum value of the critical current density was obtained for the sample with an annealing time of 270 h and a CdO nanoparticle content of 0.01.

phase stabilizer but also cause some improvement in connectivity between the grains. So, the maximum J_c was found for $Bi_{1.64}Pb_{0.36}Sr_2Ca_{1.99}Cd_{0.01}Cu_3O_y$, annealed for 270 h. When the amount of CdO is more than 0.01, the amount of unwanted phases, such as Bi-2212 and Bi-2201, increases in the sample. These unwanted phases play the role of weak links and decrease the critical current density. It is shown that the Bi-2201 phase locates mainly between the superconducting grains, preventing the supercurrent flow. It was also shown that J_C did rise as the Bi-2212 phase decreased in Bi-2223 tapes [16, 30, 31].

4 Conclusion

The substitution of Cd^{+2} for Ca^{+2} in $Bi_{1.64}Pb_{0.36}Sr_2Ca_{2-x}Cd_xCu_3O_y$ superconductor was investigated. The role of CdO nanoparticles, ac magnetic field, and annealing times on the structure and magnetic properties of $Bi_{1.64}Pb_{0.36}Sr_2Ca_{2-x}Cd_xCu_3O_y$ superconductors was studied by using the XRD and ac magnetic susceptibility techniques. The samples of $Bi_{1.64}Pb_{0.36}Sr_2Ca_{2-x}Cd_xCu_3O_y$ with $x = 0.0$, 0.01, 0.02, 0.03, 0.04, and 1 were prepared using a solid state reaction method with different annealing times. The results of XRD show that the percentage of Bi-2201 and Bi-22212 phases of the sample of $x = 0.01$ will be decreased. It was found that the Bi-2201 and Bi-2212 phases on the grain boundaries reduce the intergranular coupling. Also, the magnetic susceptibility results reveal that by increasing ac magnetic field, the peak of χ'' shifts to lower temperature. The magnetic susceptibility measurements for the samples show that the diamagnetic fraction and intergranular coupling of

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