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Absence of a pair-breaking mechanism in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$

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A high temperature $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ ($y=0$, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5) superconductor with four ZnO_2 planes is synthesized, in which the zero resistivity critical temperature $[T_c(R=0)]$, the quantity of diamagnetism, and critical current density (J_c) are found to increase with enhanced Zn doping. From the x-ray diffraction studies, the c-axis lengths are found to decrease with increased Zn doping in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3(Cu_{4-y}Zn_y)O_{12-\delta}$. The softening of apical oxygen modes observed in Fourier transform infrared absorption measurements has shown that the decreased c-axis length is among CuO_2/ZnO_2 planes. The decreased c-axis length increases the Fermi vector k_F, the coherence length ξ_c, and the superconductivity order parameter, which result into enhanced superconducting properties. The superconducting properties in these compounds were further enhanced by optimizing the carriers in CuO_2/ZnO_2 planes. The optimum numbers of carriers in these compounds were achieved by postannealing the samples in oxygen atmosphere.

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I. INTRODUCTION

The substitution of impurity atoms in high T_c cuprates (HTSCs) has been used as a probe for the study of underlying mechanism of superconductivity.[1–21] In previous studies, the magnetic impurities substituents such as Ni^{+2} at CuO_2 planar sites and Co and Fe at the chain sites of the unit cell have been found to suppress the superconducting properties.[22–27] The doping of nonmagnetic impurity atoms such as Zn^{+2} at the CuO_2 planar sites has also been found to decrease the critical temperature $[T_c(R=0)]$ in all the families of HTSCs.[1–27] The suppression of T_c in anisotropic d-wave HTSCs with the doping of Zn^{+2} atoms at CuO_2 planar sites was quite astonishing and suggested to be originating from the pair-breaking mechanism or electronic localization induced by dopant atoms.[28–51] In $YBa_2Cu_3O_{7-\delta}$ superconductor,[34] the localization of the carriers at Cu^{+2} atoms is in the immediate vicinity of Zn^{+2} atoms which was suggested to be one of the main root causes of depression of critical temperature. The suppression of superfluid density has also been observed in infrared conductivity experiments in Zn doped $YBa_2Cu_{4-x}Zn_xO_8$ single crystals and a decrease of $T_c(R=0)$ in part was suggested to be associated with the localization of carriers.[38] We have recently observed enhanced superconductivity by Zn doping in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ ($y=0$, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5) superconductors, which is quite contrary to all the previous studies on Zn doped cuprate HTSCs. In these Zn doped superconductors, we have also tried to explore the possibility of existence of localization of carriers at Cu^{+2} and /or Zn^{+2} sites by carrying out postannealing experiments in nitrogen and oxygen atmospheres. Through these studies, we have observed that there is no localization of the carriers to the Cu^{+2} or Zn^{+2} sites in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors. In the light of the present results, we have looked into the possible reasons for the suppression of superconductivity by Zn doping in all previous studies.

II. EXPERIMENT

The $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ ($y=0$, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5) samples were prepared by the solid state reaction method accomplished in two stages. At the first stage, $Cu_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ precursor material was prepared by thoroughly mixing ZnO, $Ba(NO_3)_2$, $Ca(NO_3)_2$, and Cu(CN) in a quartz mortar and pestle in appropriate ratios. The mixed material was fired twice at 880 °C in a quartz boat for 24 h and furnace cooled to room temperature. At the second stage, the precursor material was ground for about an hour and mixed with Tl_2O_3 to give $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ as the final reactant composition. Thallium mixed material was pelletized under 3.8 $tons/cm^2$ and the pellets were enclosed in a gold capsule. Gold capsule containing pellets were annealed for about 10 min at 880 °C followed by quenching to room temperature. The samples were characterized by resistivity, ac susceptibility, and critical current density measurements. The structure of the material was determined by x-ray diffraction scan from Rigaku D/Max IIIC using a Cu $K\alpha$ source with wavelength of 1.540 56 Å and the cell parameters were determined by a cell refinement computer program. The phonon modes related to the vibrations of various oxygen atoms in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ unit cell were observed by Nicolet 5700 Fourier transform infrared (FTIR) spectrometer in the 400–650 cm^{-1} wave number range. The postannealing of the samples was carried out at 550 °C for 6 h. This temperature is chosen because oxygen intercalation into the unit cell is observed around this temperature regime. The oxygen content of the samples were determined by standard iodometric titration method.[52–54]

III. RESULTS AND DISCUSSION

The x-ray diffraction scan of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_1Zn_3O_{12-\delta}$ superconductor is shown in Fig. 1 in the inset of this figure, a decrease in the c-axis lengths with the increased Zn doping is shown. Most of the diffraction lines are fitted following the $P4/mmm$ space group and tetragonal structure. The decreased c-axis length with the increased Zn concentration shows an improved interplane coupling, which is consistent with previous studies carried out on Zn doped samples.[31] The effect of decreased

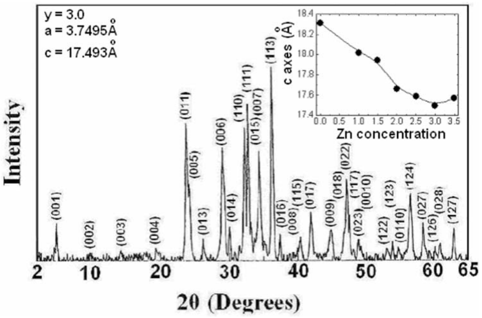

FIG. 1. The XRD pattern of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_1Zn_3O_{12-\delta}$ superconductor (inset c-axes vs Zn concentration).

c-axis length can also be seen in the form of softening of phonon modes[55–60] of apical oxygen atoms in Zn doped $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_1Zn_3O_{12-\delta}$ samples. In Zn-free samples ($y=0$), the phonon modes related to apical oxygen atoms[61–64] of type Tl-O_A-Cu(2) and Cu(1)-O_A-Cu(2) are observed around 501 and 537 cm^{-1}, which are softened in all Zn doped samples to 453–455 and 514–524 cm^{-1}, respectively (Fig. 2). These two modes are of Tl-O_A-Cu(2)/Zn and Cu(1)-O_A-Cu(2)/Zn types. If the softening of these apical oxygen modes is linked with the decreased c-axis lengths observed by x-ray diffraction (XRD) measurements, then it becomes evident that the bond distances along the c axis are decreased between the CuO_2/ZnO_2 planes. The planar mode of oxygen atoms in Zn free samples ($y=0$) is observed around 575 cm^{-1}, which is softened in Zn doped $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_1Zn_3O_{12-\delta}$ samples to 572–565 cm^{-1}. The softening of planar oxygen mode is most likely arising from the increased mass of Zn (65.38 amu) with respect to Cu (63.546 amu).

The resistivity measurements of Zn doped $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ ($y=0$, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5) samples are shown in Fig. 3. It can be seen that the magnitude of normal state resistivity decreases and $T_c(R=0)$ increases with the increased Zn concentration in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ unit cell. The bulk superconductivity in the samples checked by ac susceptibility measurements are shown in Fig. 4; for a relative comparison of magnitude of diamagnetism in all Zn doped samples, we have normalized the ac signal to the mass of the sample. The onset of the superconductivity, magnitude of diamagnetism, and peak temperatures (T_P) observed in the out of phase component of magnetic ac susceptibility are found to increase systematically with the increased Zn doping in $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ samples (Fig. 5). T_P is related to H_c (critical magnetic field) and hence to the critical current density (J_c) of the samples.[65,66] The increase in T_p with increased Zn doping suggested that J_c is enhanced with Zn doping, which is also observed in transport J_c measurements, as discussed in the next section. In the previous studies on Zn doped HTSC systems, $T_c(R=0)$ and magnitude of diamagnetism are significantly suppressed by enhanced Zn^{+2} doping in the final compound. The localization of free carriers at Cu^{+2} sites in the neighborhood of Zn^{+2} atoms was suggested to be the main route cause of $T_c(R=0)$ suppression in those studies. Those Cu^{+2} atoms having a localization of carriers in their neighborhoods behave as efficient scattering centers,[28–51] which are absent altogether in our $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors.

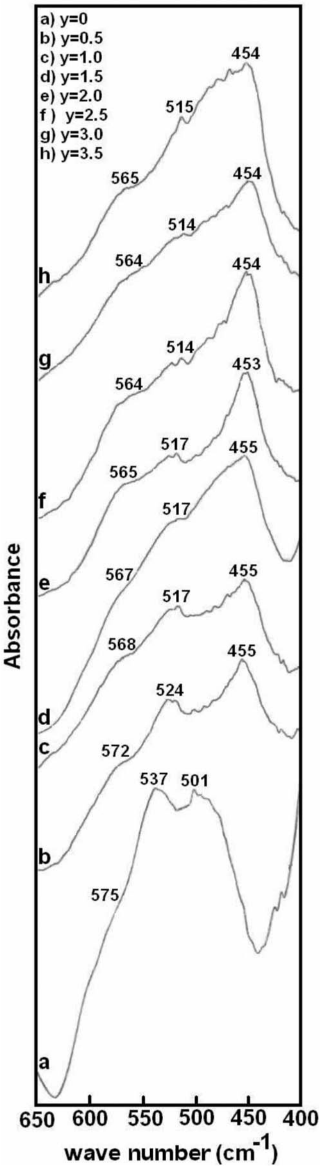

FIG. 2. The FTIR absorption spectra of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors.

The possibility of existence of localization of carriers to the Cu^{+2} sites in the neighborhood of Zn^{+2} atoms, if present, could be enhanced by increasing and/or decreasing the carrier concentration in CuO_2/ZnO_2 planes. In order to change the carrier concentration in CuO_2/ZnO_2 planes, we have carried out postannealing experiments at 550 °C in oxygen, air, and nitrogen atmospheres. For such studies, we have chosen the samples without Zn ($y=0$) and with maximum Zn doping ($y=3.5$). $T_c(R=0)$, T_p, I_c, and oxygen content for these experiments are summarized in Fig. 6. Since oxygen has the second highest electronegativity in the Periodic Table of elements, the intercalation of it in the $Cu_{0.5}Tl_{0.5}Ba_2O_{4-\delta}$ charge reservoir layer can efficiently dope the charge carriers to conducting CuO_2/ZnO_2 planes. The higher oxygen content

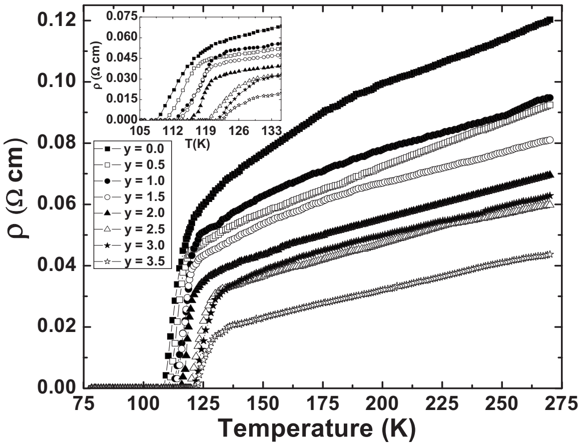

FIG. 3. The resistivity vs temperature of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors.

in the charge reservoir layer decreases, whereas their lower concentration increases the electron density in conducting planes; the former enhances, while the later decreases the number of holes in ZnO_2/CuO_2 planes. $T_c(R=0)$, T_p, I_c, and oxygen content of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ ($y=0$, 3.5) samples are increased after postannealing in oxygen, whereas these parameters are suppressed by postannealing in air and nitrogen atmospheres. The increased oxygen content in the samples after postannealing in oxygen led to a suggestion that the more intake of oxygen in $Cu_{0.5}Tl_{0.5}Ba_2O_{4-\delta}$ charge reservoir layer brings the concentration of electrons in the conducting ZnO_2/CuO_2 planes to an optimum level, which consequently reduce the electron hole recombination processes at lower temperatures and an enhancement in the population of holes in conducting planes is promoted. The enhanced carrier's density in ZnO_2/CuO_2 planes promotes an increase in the Fermi vector $k_F=(3\pi^2N/V)^{1/3}$, the coherence length along the c axis $\xi_c=(\hbar k_F/2m\Delta)$, and the Fermi velocity $v_F=(\pi\xi_c\Delta/\hbar)$ of the carriers,[67,68] which ultimately enhance the superconductivity parameters.

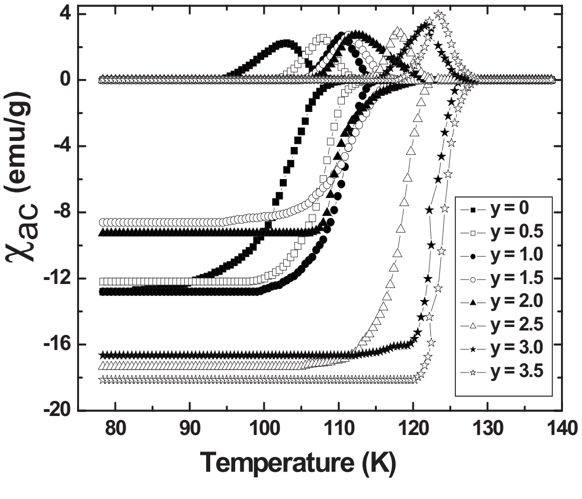

FIG. 4. The ac susceptibility vs temperature of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors.

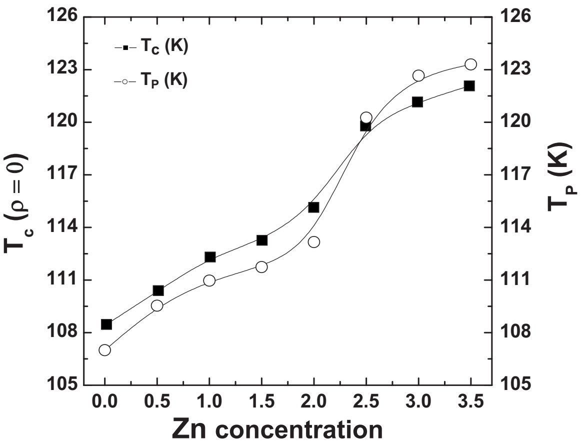

FIG. 5. $T_c(R=0)$ and T_p in out of phase component of magnetic susceptibility vs Zn concentration of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors.

These self-doping experiments were performed to determine unevoqivocal evidence of the role played by doped carriers in ZnO_2/CuO_2 planes in the mechanism of superconductivity in these Zn doped compounds. As proposed in the previous studies that Zn doping promotes localization of carriers to the Cu^{+2} sites in the immediate vicinity of Zn^{+2} atoms. It was expected that increased hole concentration in ZnO_2/CuO_2 planes by the postannealing in oxygen would have enhanced such proposed localization processes. To our surprise, no such localization has been observed; otherwise, it would have killed the superconductivity; instead, it is enhanced. These experiments lead us to a conclusion that the role of the doped carriers in the conducting planes is primary and their localization at Cu^{+2}/Zn^{+2} sites is absent altogether in our $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{0.5}Zn_{3.5}O_{12-\delta}$ superconductor. The decreased hole concentration as a result of postannealing in air and nitrogen makes the superconductor inferior but no localization of carrier is witnessed; otherwise, superconductivity would have been destroyed. These studies also suggested that the role of the ratio of divalent/trivalent (Cu^{+2}/Tl^{+3}) atoms in $Cu_{0.5}Tl_{0.5}Ba_2O_{4-\delta}$ charge reservoir layer is primary in supplying the charge carriers to the CuO_2/ZnO_2 planes.

The question arises that why in the previous studies the superconductivity is suppressed by Zn doping in all cuprate HTSC systems?[28–51] To answer this question, we have replaced $Cu_{0.5}Tl_{0.5}Ba_2O_{4-\delta}$ charge reservoir layer in our $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{0.5}Zn_{3.5}O_{12-\delta}$ superconductor by $TlBa_2O_{4-\delta}$ and synthesized $TlBa_2Ca_3Cu_{0.5}Zn_{3.5}O_{12-\delta}$ material. Our $TlBa_2Ca_3Cu_{0.5}Zn_{3.5}O_{12-\delta}$ system is identical in the composition of the charge reservoir layer with Tl^{+3} in it; the other cuprate HTSC systems have trivalent atoms such as La^{+3}, Y^{+3}, Bi^{+3}, etc., in their charge reservoir layers. To our surprise, $TlBa_2Ca_3Cu_{0.5}Zn_{3.5}O_{12-\delta}$ material has not shown signs of superconductivity down to 77 K. These results are identical and consistent with the previous studies on Zn doped LaBaCuO, $YBa_2Cu_3O_y$, and BiSrCaCuO superconductors. If we carefully look into these compounds, we would see that these materials had trivalent ions (i.e., La^{+3}, Y^{+3}, and Bi^{+3}) either in the charge reservoir layer or in be-

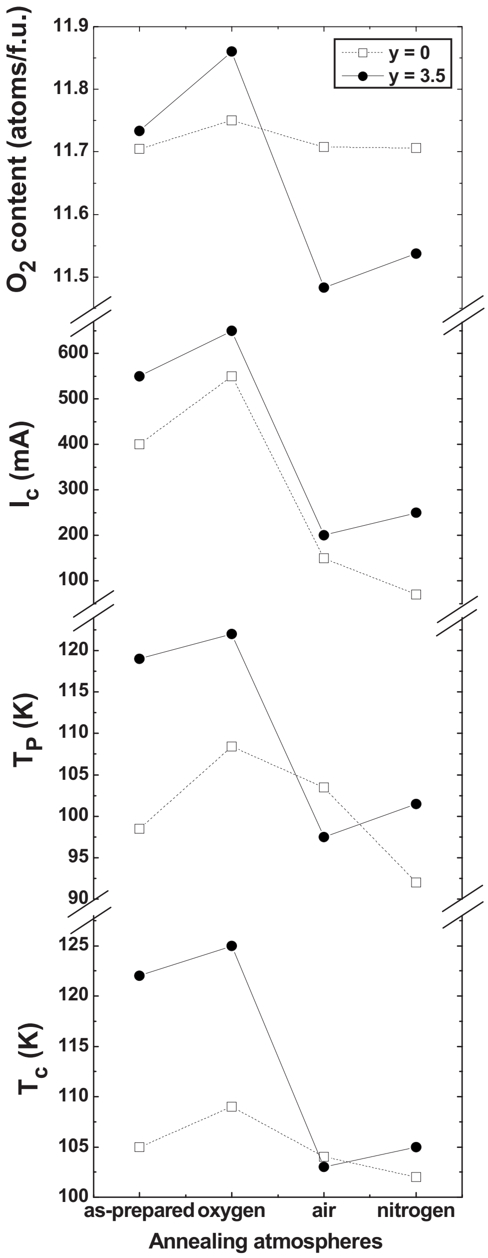

FIG. 6. $T_c(R=0)$, T_p, I_c, and oxygen contents vs annealing atmospheres of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors.

tween the CuO_2 planes. The presence of trivalent ions, most likely localize the carriers in their neighborhood and their localization at the Cu^{+2} sites in the vicinity of Zn^{+2} atoms, as suggested in previous studies, is unlikely. These experiments have also shown that when 100% trivalent ions are present in the charge reservoir layers, it is most likely incapable of supplying carriers to the conducting ZnO_2 planes and superconductivity is destroyed.

IV. CONCLUSIONS

In conclusion, we have successfully synthesized Zn doped $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ ($y=0$, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5) superconductors, in which $T_c(R=0)$ and the quantity of diamagnetism are enhanced with the increased Zn doping. These superconductors have tetragonal crystal structure and their c-axis length decreases with increased Zn concentration in the unit cell. The decreased c-axis length is also supported by the softening of apical oxygen phonon modes. The decreased c-axis length decreases volume of the unit cell which in turn increases the Fermi vector $k_F=(3\pi^2 N/V)^{1/3}$, the coherence length along the c axis $\xi_c=(\hbar k_F/2m\Delta)$, and the Fermi velocity $v_F=(\pi\xi_c\Delta/\hbar)$ of the carriers,[67,68] which ultimately lowers the anisotropy of the final compound. The enhanced ξ_c and v_F most likely enhance the $T_c(R=0)$, magnitude of diamagnetism, and J_c in Zn doped samples. The postannealing in oxygen has shown increased superconducting properties, which may possibly be arising from the optimization of carriers in ZnO_2 planes. It is worth emphasizing that the charged carriers in case of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors are most likely supplied by the ratio of Cu^{+2}/Tl^{+3} in the $Cu_{0.5}Tl_{0.5}Ba_2O_{4-\delta}$ charge reservoir layers. The presence of Cu^{+2} in the $Cu_{0.5}Tl_{0.5}Ba_2O_{4-\delta}$ charge reservoir layers of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ most efficiently dopes the CuO_2/ZnO_2 planes with carriers and we get enhanced superconductivity. This is contrary to the Zn doped $La_{2-x}Sr_xCu_{1-y}Zn_yO_4$, $YBa_2Cu_{3-x}Zn_xO_{7-\delta}$, $YBa_2Cu_{4-x}Zn_xO_8$, $Bi_2Sr_2Ca_{1-x}Y_x(Cu_{1-y}Zn_y)_2O_{8+\delta}$, and $TlBa_2Ca_2Cu_3O_{9-\delta}$ superconductors, where the presence of M^{+3} ($M=La,Y,Bi,Tl$) possibly had suppressed the critical temperature. These studies strongly suggested that the proposed localization of the carriers by Zn doping in all previous studies[28–51] was most likely at the trivalent M^{+3} sites and not at the divalent Cu^{+2} sites. It is worth emphasizing that zinc oxides belong to wide band gap semiconductors with energy gap of 3.2 eV,[69] the existence of superconductivity in mixed CuO_2 and ZnO_2 planes could be a key to the understanding of mechanism of superconductivity since we very well know the properties of semiconductors. Moreover, the ZnO-based semiconductors could be transformed into p type by doping Ge and n type by doping Si; therefore, we can make electron and hole doped superconductors from $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors. The preparation of $Cu_{0.5}Tl_{0.5}Ba_2Ca_3Cu_{4-y}Zn_yO_{12-\delta}$ superconductors by this method is extremely reproducible and the detailed characterization of it is underway.

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