

# Infrared absorption spectroscopy and fluctuations induced conductivity (FIC) analysis of Be-doped $\text{TiBa}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$ superconductor

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## Abstract

Be-doped  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  ( $y=0, 0.25, 0.5, 0.75$ , and  $1.0$ ) superconductor bulk samples were synthesized by solid state reaction and characterized by X-ray diffraction (XRD), dc-resistivity  $\{\rho \text{ (}\Omega \text{ cm)}\}$ , and Fourier Transform Infrared (FTIR) absorption spectroscopy. Fluctuations induced conductivity (FIC) analysis is carried out on temperature dependent dc-resistivity data of as-prepared and oxygen post-annealed  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  superconductor samples by using Aslamazov–Larkin (AL) and Lawrence–Doniach (LD) models for excess conductivity. Different microscopic parameters such as zero temperature coherence length along  $c$ -axis  $\{\xi_c(0)\}$ , inter-layer coupling ( $J$ ), inter-grain coupling ( $\alpha$ ), critical exponent ( $\lambda_D$ ) and dimensionality of fluctuations are calculated for understanding the role of Be-doping on superconducting properties of  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  samples. The cross-over temperature ( $T_o$ ) is shifted towards higher temperature values with the increase of Be contents in  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  samples. The increase in  $\xi_c(0)$  and  $J$  after Be-doping at Ca sites shows the improvement of inter-plane coupling in  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  samples. The increase in zero resistivity critical temperature  $\{T_{c(R=0)} \text{ (K)}\}$  up to  $y=0.5$  and then decrease for  $y=0.75, 1.00$  fixed the Be-doping level for optimum increase of superconducting properties of  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  samples. The appreciable changes in all the microscopic parameters extracted from the FIC analysis and the increase in relative intensity of almost all the oxygen phonon modes indicate the oxygen diffusion in the unit cell after oxygen post-annealing the samples. The oxygen diffusion can take place at both inter-granular and intra-granular sites, which increase the superconducting volume fraction by improving the grains size, inter-grain connectivity and carrier density.

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## 1. Introduction

The small coherence lengths and large anisotropies of cuprates are responsible for thermal fluctuations, which are observable in several temperature dependent transport properties [1–5]. Many experimental [6–8] as well as theoretical [9,10] results were reported regarding the critical effects arising from thermal fluctuations in high  $T_c$  superconductors (HTSCs). The experimentally observed rounding of the resistivity curves has been interpreted frequently as a possible signature of onset of high  $T_c$  superconducting phase. As a result of thermal fluctuations, there is a finite probability of

Cooper-pair formation above  $T_c^{\text{onset}}$  (K), which gives rise an excess conductivity [1]. It is now thought that the mechanism of superconductivity in cuprates can be somehow understood by studying their normal-state properties, which are extremely specific [11–14]. However, in spite of the significant efforts of researchers, the physics of superconducting pairing and the mechanisms for the scattering of carriers in the normal state are not yet completely clear. Even the results of such classic experiments as resistivity measurements and Hall effect are extremely contradictory [15]. Thus the study of FIC can give useful informations about the scattering and fluctuational pairing mechanisms as  $T$  approaches  $T_c^{\text{onset}}$  (K). The equation determining FIC is conveniently written as

$$\Delta\sigma(T) = \left[ \frac{\rho_n(T) - \rho(T)}{\rho_n(T)\rho(T)} \right] \quad (1)$$

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where  $\rho(T)$  is the actually measured resistivity, and  $\rho_n(T) = \alpha + \beta T$  is the normal-state resistivity of the sample extrapolated to the resistivity at 0 K. This definition of  $\rho_n(T)$  is widely used for calculating  $\Delta\sigma(T)$  in HTSCs [16–21] in spite of very long dispute especially in under-doped systems for which the deviation of  $\rho(T)$  from its linear dependence is observed at very high temperature before  $T_c^{\text{onset}}$  (K). To obtain the information about the mechanisms for scattering and superconducting fluctuational pairing in cuprates, we have analyzed the temperature dependent dc-resistivity data of Be-doped  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  samples. There are two fluctuation contributions to  $\Delta\sigma(T)$ ; one direct contribution, which was given a theoretical foundation by Aslamazov and Larkin (AL) [22], arises as a result of the spontaneous formation of fluctuation-induced Cooper pairs above  $T_c^{\text{onset}}$  (K). The second contribution introduced by Maki and Thompson (MT) [23,24] in an extension of the AL theory is interpreted as a result of an interaction of already existing fluctuational pairs with normal charge carriers and is governed by pair-breaking processes. The MT contribution depends on the life time of the fluctuational pairs and is dominant in two-dimensional (2D) region of fluctuations [25], especially in well-structured samples, i.e. in the case of weak pair-breaking, whereas the AL mechanism dominates in three-dimensional (3D) region of FIC near  $T_c^{\text{onset}}$  (K). In layered structures, the AL contribution is usually dominated by the Lawrence–Doniach (LD) model [26], which predicts a smooth dimensional cross-over from 2D to 3D fluctuation region for  $T \rightarrow T_c^{\text{onset}}$  (K). Here the MT contribution is assumed to be insignificant and the question of a change-over of fluctuation mechanisms does not arise. The FIC according to AL theory is given as

$$\Delta\sigma(T) = A\varepsilon_D^{-\lambda} \quad (2)$$

where  $A$  is the fluctuation amplitude,  $\lambda_D$  is the dimensional exponent, and  $\varepsilon = \ln[T/T_c^{\text{mf}}]$  is the reduced temperature and  $T_c^{\text{mf}}$  is usually referred as the mean field critical temperature [15,27–30]. The temperature, which separates the mean field region from the critical region, is known as  $T_c^{\text{mf}}$ . The exponent  $\lambda_D$  determines the dimensionality of the superconducting thermal fluctuations and is given as  $\lambda_D = 2 - D/2$ ;  $\lambda_D = 1/2, 1, 3/2$  for three-, two- and one-dimensional fluctuations, respectively. There is one more region of fluctuations close to critical temperature, which is called the critical region; the exponent for the critical region [31–33] is  $\lambda_D = 1/3$ . In the present paper, we focused on the fluctuations in the mean field region. The accurate determination of  $T_c^{\text{mf}}$  is very important to study the fluctuations in mean field region. The MT term becomes important in the region of moderate pair-breaking [15,25–27]. The MT contribution in cuprates is over-damped due to the strong phase breaking, and is not usually observed [34]. The LD model is a modified form of AL theory, which predicts a cross-over from two-dimensional (2D) conductivity to three-dimensional (3D) conductivity at a cross-over temperature

$$T_o = T_c \left[ 1 + \left( \frac{2\xi_c(0)}{d} \right)^2 \right] \quad (3)$$

In layered superconductors at very low temperature close to zero resistivity temperature  $\xi_c > d$ ; where  $d$  is the distance between the charge reservoir layers of unit cell ( $d$  is

approximately equal to the  $c$ -axis lattice parameter of the unit cell). At the cross-over temperature, the system transforms to 3D fluctuations region. The 3D region persists as long as the Josephson coupling between the  $\text{CuO}_2$  planes is possible. In the following section, we have discussed the FIC of  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  superconductor in the mean field region using the AL theory. We did not observe MT type contribution to the FIC in all these samples as was expected. The motivation of this study is to investigate the role of Be-doping at Ca sites in between the  $\text{CuO}_2$  planes as well as insulating charge reservoir layer of  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  samples on superconductivity. For this purpose FIC analysis has been carried out on temperature dependent resistivity data. Thus, FIC analysis provides the reliable informations about the zero temperature coherence length along the  $c$ -axis  $\xi_c(0)$ , phase relaxation time ( $\tau_\phi$ ), and the conduction dimensionality of the carriers in cuprates.

## 2. Experimental details

The superconducting  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  ( $y=0, 0.25, 0.5, 0.75$ , and  $1.0$ ) samples were prepared by the solid state reaction accomplished in two stages. At the first stage,  $\text{Ba}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  precursor material was prepared by thoroughly mixing of  $\text{Ba}(\text{NO}_3)_2$ ,  $\text{Ca}(\text{NO}_3)_2$ ,  $\text{BeO}$  and  $\text{Cu}(\text{CN})$  compounds of purity 99.99% in a quartz mortar and pestle in appropriate ratios. The mixed material loaded in a quartz boat was fired twice at  $860^\circ\text{C}$  for 24 h in a preheated furnace. After 24 h heat treatment, the furnace was switched off and the precursor material was cooled down to room temperature. At the second stage, thallium oxide ( $\text{Tl}_2\text{O}_3$ ) was mixed with precursor material and was ground for about an hour. The material mixed with  $\text{Tl}_2\text{O}_3$  was palletized under  $3.8 \text{ tons/cm}^2$  pressure and the pellets were enclosed in gold capsules. The pellets enclosed in gold capsules were sintered at  $860^\circ\text{C}$  for about 10 min in preheated furnace and then quenched to room temperature. The structure of the material was determined by XRD scans from Brouker X-ray diffractometer using  $\text{CuK}\alpha$  source of wavelength  $1.54056 \text{ \AA}$  and cell parameters were calculated by using the cell refinement computer program. The temperature dependent dc-resistivity of the samples was measured by conventional four-probe method from room temperature (290 K) to liquid nitrogen temperature (77 K). The various oxygen phonon modes were studied by FTIR absorption spectroscopy. The post-annealing of the samples in flowing oxygen was carried out in a tubular furnace at  $500^\circ\text{C}$  for 6 h.

## 3. Results and discussion

The XRD scans of  $\text{TiBa}_2(\text{Ca}_{2-y}\text{Be}_y)\text{Cu}_3\text{O}_{10-\delta}$  ( $y=0, 0.25, 0.5$ , and  $0.75$ ) are shown in Fig. 1. Most of the XRD peaks are indexed according to the tetragonal structure following  $\text{P4}/\text{mmm}$  space group with cell parameters  $a=4.22 \text{ \AA}$  and  $c=15.20 \text{ \AA}$  for the samples with  $y=0$  and  $a=4.22 \text{ \AA}$  and  $c=15.11 \text{ \AA}$ ,  $a=4.13 \text{ \AA}$  and  $c=14.97 \text{ \AA}$ ,  $a=4.11 \text{ \AA}$  and  $c=14.90 \text{ \AA}$  for the samples with  $y=0.25, 0.5$ , and  $0.75$ ,

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