

# Effect of La Doping on Structural and Superconducting Properties of (Bi, Pb)-2223 Ceramics

Mounira HAMEL<sup>1</sup>, Faiza BOUAÏCHA<sup>2,3\*</sup>, Sonia ATTAF<sup>1,4</sup>,  
Mohamed-Fayçal MOSBAH<sup>1</sup>

<sup>1</sup> Physics Department, Material Science and Applications Research Unit, University of Mentouri, Constantine 1, Road of Ain-El-Bey, 25017 Constantine, Algeria

<sup>2</sup> Laboratory of the Active Components and Materials, University Larbi Ben M'hidi, B.P 358 Road of Constantine 04000, Oum El Bouaghi, Algeria

<sup>3</sup> Physical Measurement Department, Institute of Technology, University Larbi Ben M'hidi, Oum El Bouaghi, Algeria

<sup>4</sup> Laboratory of Ceramics, Constantine University, 1 Road of Ain El Bey 25000 Constantine, Algeria

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Superconducting ceramics with nominal composition  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+\delta}$  ( $0 \leq y \leq 0.04$ ); were prepared in pellet form by conventional solid-state reaction method. The samples were characterized by powder X-ray diffraction (XRD), scanning electron microscopy (SEM), temperature-dependent resistivity measurements, and AC susceptibility. XRD patterns showed the coexistence of the Bi-2223 and Bi-2212 phases, with all samples exhibiting an orthorhombic structure. With increasing La concentration, the b lattice parameter increased, whereas the c lattice parameter decreased. These changes are consistent with a structural modification associated with La incorporation and may be related to the larger ionic radius of  $\text{La}^{3+}$  compared with  $\text{Ca}^{2+}$  and to changes in the oxygen distribution between the  $\text{CuO}_2$  and BiO planes. Electrical resistivity measurements showed that the superconducting transition temperature increased up to  $y = 0.01$  and decreased for higher La concentrations, while the residual resistivity increased above  $y = 0.01$ , suggesting increased structural or compositional inhomogeneity.

**Keywords:** superconductivity, SHTC, Bi-Pb-Sr-Ca-Cu-O system, substitution, resistivity, normal state and Bi-2223.

## 1. INTRODUCTION

The BSCCO (Bi-Sr-Ca-Cu-O) system is one of the most studied high-temperature superconductors, with a critical temperature ( $T_c$ ) ranging from 7 K to 110 K depending on the crystalline phase [1, 2]. The major difference between these phases of this system lies in the number of  $\text{CuO}_2$  and Ca layers present in their crystal structure. Some of the most important phases are Bi-2201, Bi-2212, Bi-2223, and Bi-2234 [1, 2, 3, 4]. The study of this system has contributed to the development of other superconductors with similar layered structures, such as the Tl-Ba-Cu-Cu-O and Hg-Ba-Cu-Cu-O systems, were developed. The (Bi, Pb)-2223 phase is the most widely used for practical applications such as long wires [5], contains three  $\text{CuO}_2$  planes, and has the highest critical temperatures of bismuth-based superconductors. The (Bi, Pb)-2223 phase is obtained with the Bi-2212 phase, and characterized by its an easier preparation and a higher volume fraction [3, 5, 6]. In this phase, the inner  $\text{CuO}_2$  plane is surrounded by calcium (Ca) planes and does not have apical oxygen as the other two  $\text{CuO}_2$  planes. Furthermore, the coupling of these planes is weaker compared to the Bi-2212 phase, and this does not appear to significantly affect the critical temperature ( $T_c$ ) [7].

In general, the critical temperature and superconductivity can decrease at a rate dependent on the

rare-earth element used for substitution. The spin or spinless state of the substituted ion does not significantly affect the substitution of metallic elements at the copper site [4]. On the other hand, the critical temperature ( $T_c$ ) can be modified by changing the charge carrier density, as occurs when calcium ions ( $\text{Ca}^{2+}$ ) are replaced by rare-earth element ions with a valence of +3, such as samarium, neodymium, and yttrium [1, 2, 8]. In this work, the effect of lanthanum doping on the (Bi, Pb)-2223 phase is investigated. The doping processes are currently used to improve the structural, electrical, and magnetic properties of oxide superconductors, with a particular focus on enhancing their performance in practical applications [7, 8, 9].

## 2. EXPERIMENT

The preparation of samples of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+\delta}$ , with ( $0 \leq y \leq 0.04$ ), was done using a conventional solid-state method that utilizes the Bi ((P, La)-2212 ( $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{(1-y)}\text{La}_y\text{Cu}_2\text{O}_{(8+\delta)}$ )) doped Lanthanum and  $\text{CaCuO}_2$  phase as precursors. A stoichiometric ratio of Bi/Pb/Sr/Ca/La/Cu/O was used to weigh the highly pure powders of  $\text{Bi}_2\text{O}_3$ , PbO,  $\text{SrCO}_3$ ,  $\text{CaCO}_3$ , CuO and  $\text{La}_2\text{O}_3$ , which is equivalent to 1.6/0.4/y/y/1/8. The powders were mixed, ground in an agate mortar, and calcined at 810 °C for 30 hours to obtain samples of undoped Bi (Pb)-2212 and doped Bi (Pb, La)-2212 phases. The  $\text{CaCuO}_2$  phase was

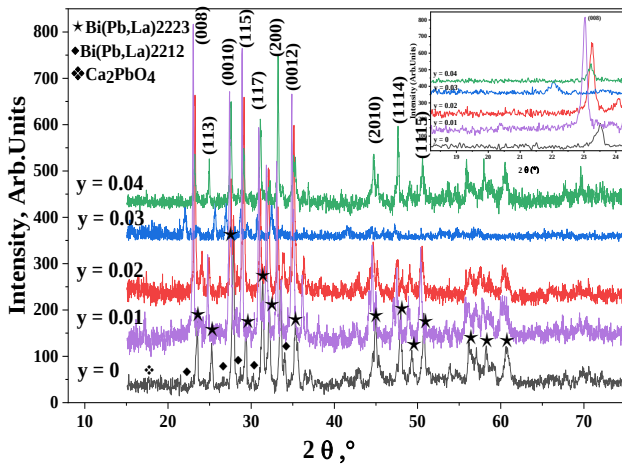
\* Corresponding author: Faiza. Bouaïcha  
E-mail: [bouaicha.faiza@univ-oeb.dz](mailto:bouaicha.faiza@univ-oeb.dz)

prepared using powders of CaO and CuO, and by sintering the ground mixture at 900 °C for 40 hours. After 12 hours of sintering at 900 °C with CaCO<sub>3</sub> powder, the CaO was obtained. The Bi (Pb)-2212 undoped and Bi (Pb, La)-2212 doped and CaCuO<sub>2</sub> calcined powders were ground, mixed, formed with a pressure of 5 ton/cm<sup>2</sup> into pellet shape and sintered for 120 h at a temperature of 850 °C. After grinding and forming into pellets, a second sintering at 850 °C for 60 hours are carried out. All these thermal treatments were performed using a heating or cooling rate of 5 °C/min.

The samples have been characterized by XRD analysis at each step of the preparation using copper K $\alpha$  radiation ( $\lambda_{CuK\alpha} = 1.5418 \text{ \AA}$ ). Diffraction spectra peaks was identified using the following references JCPD-ICDD database [10]: JCPDS 80-2029 for Bi-2212, JCPDS 39-0283 for Bi-2201, JCPDS 80-2030 and JCPDS 42-0450 for Bi-2223, and JCPDS 76-2128 for Ca<sub>2</sub>PbO<sub>4</sub>. Using CELREF V3 software, lattice parameters were calculated. The tilt angle of zero was utilized to analyze grain morphology using TESCAN scanning electron microscopy. A classical four-point technique was utilized to measure electrical resistivity, which involved an AC current with a constant amplitude of 1 mA and a frequency of 18 Hz. Using a zero field cooled mode from 5 to 120 K, the Quantum Design SQUID magnetometer was used to measure AC susceptibility.

### 3. RESULTS AND DISCUSSION

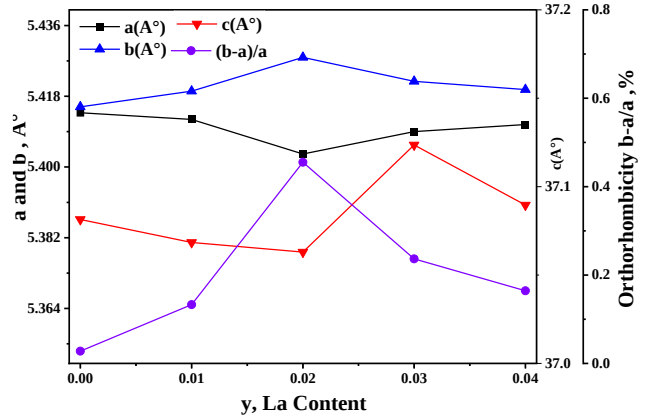
XRD patterns (Fig. 1) of undoped and doped Bi (Pb, La)-2223 samples revealed that the majority of the peaks were indexed in the Bi (Pb, La)-2223 phase, symbolized by ( $\star$ ), with traces of the secondary phases Bi (Pb, La)-2212 and Ca<sub>2</sub>PbO<sub>4</sub>, respectively, are symbolized by: ( $\diamond$ ) and ( $\blacklozenge$ ). All samples confirmed the presence of (Ca<sub>2</sub>PbO<sub>4</sub>), identified by its characteristic peak located at  $2\theta = 17.68$  [11]. the upper right insert displays the angular position due to the lanthanum content.



**Fig. 1.** XRD patterns of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+d}$ , with  $0 \leq y \leq 0.04$ . The insert on the top right shows variation in the position of 008 peaks' reflections.

All prepared samples were indexed on an orthorhombic symmetry according to Vivian Delmoute Rodrigues et al. [12] and A. Sedky et al. [13]. The displacement is on the left side indicating a reduction of the reticular length. When the

Lanthanum content is lowest, the displacement is highest, and it always goes to a mean position on the left side. Fig. 2 shows the evolution of the lattice parameters ( $a$ ), ( $b$ ), and ( $c$ ) together with the orthorhombic distortion as a function of the La content. Concurrently, the cell parameters  $a$  and  $b$  exhibit a minimum and maximum, respectively.



**Fig. 2.** Variation of  $a$ ,  $b$ , and  $c$  lattice parameters and orthorhombicity  $b-a/a$  of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+d}$  samples with  $y$  of La content

As the lanthanum content increases from 0 to 0.04, the parameter  $a$  decreases from 5.4138 Å for the undoped sample to 5.4033 Å at  $y = 0.02$ , slightly while  $b$  increases from 5.4153 Å to 5.4279 Å. Consequently, the difference between  $a$  and  $b$  increases, leading to a progressive enhancement of the orthorhombic distortion, which reaches its maximum at  $y = 0.02$ . For higher La contents, the  $a$  and  $b$  parameters tend to approach each other, resulting in a decrease in the orthorhombic distortion. This results in an anisotropic distortion of the crystal lattice, linked to the replacement of  $\text{Ca}^{2+}$  by  $\text{La}^{3+}$ . This conclusion is reasonable since the ionic radius of Ca (0.99 Å) is smaller than that of La (1.06 Å) [13]. On the other hand, the  $c$ -parameter initially decreases from 37.0814 Å for  $y = 0$  to 37.0630 Å for  $y = 0.02$ , then increases again to reach values higher than those of the undoped sample.

The orthorhombic distortion was estimated from the lattice parameters using the relation:  $\text{OD} = \frac{b-a}{a}$ , following the methodology described in the study of lanthanum-substituted (Bi, Pb)-2223 superconductors [14]. The calculated orthorhombic distortion value increases from 0.0277 % for the undoped sample to 0.0455 % at  $y = 0.02$ , then decrease for higher lanthanum concentration. Thus, a maximum orthorhombic distortion is observed at  $y = 0.02$ , indicating that lanthanum substitution significantly modifies the structural distortion of the Bi (Pb, La)-2223 phase. However, this was related to the amount of the transfer excess oxygen from  $\text{La}_2\text{O}_3$  during replacing  $2\text{CaO}$  one in the  $\text{BiO}$  planes and the length of the Cu-O bond in the Cu-O<sub>2</sub> planes, as reported elsewhere [2, 15–17]. The increased oxygen content strengthens the bonding, resulting in a reduction of the lattice parameter  $c$  and an increase of the orthorhombic distortion. Table 1 summarized the lattice parameters  $a$ ,  $b$  and  $c$ , and orthorhombic distortion of the samples doped with lanthanum. For the qualitative analysis, the relative volume fractions of the Bi (Pb, La)-2223, Bi (Pb, La)-2212, and Ca<sub>2</sub>PbO<sub>4</sub> phases were determined using by Eq. 1 [18, 19]:

**Table 1.** The lattice parameters and orthorhombicity of the Bi (Pb, La)-2223samples

| Content y of La | Orthorhombic unit cell Bi (Pb, La)-2223 phase |                 |                  | Orthorhombic distortion<br>OD = (b-a)/a, % |
|-----------------|-----------------------------------------------|-----------------|------------------|--------------------------------------------|
|                 | a, Å                                          | b, Å            | c, Å             |                                            |
| 0               | 5.4138 ± 0.0670                               | 5.4153 ± 0.0749 | 37.0814 ± 0.4990 | 0.0277 ± 1.856                             |
| 0,01            | 5.4121 ± 0.0065                               | 5.4193 ± 0.0075 | 37.0684 ± 0.0421 | 0.1330 ± 0.183                             |
| 0,02            | 5.4033 ± 0.0051                               | 5.4279 ± 0.0048 | 37.063 ± 0.0313  | 0.4553 ± 0.130                             |
| 0,03            | 5.4090 ± 0.0117                               | 5.4218 ± 0.0101 | 37.1236 ± 0.0624 | 0.2366 ± 0.286                             |
| 0,04            | 5.4108 ± 0.0028                               | 5.4197 ± 0.0038 | 37.0896 ± 0.0179 | 0.1645 ± 0.087                             |

$$V_{\text{Bi(Pb,La)2223}}\% = \frac{\sum I_{\text{Bi(Pb,La)2223}}}{\sum I_{\text{Bi(Pb,La)2223}} + \sum I_{\text{Bi(Pb,La)2212}} + \sum I_{\text{Ca}_2\text{PbO}_4}};$$

$$V_{\text{Bi(Pb,La)2212}}\% = \frac{\sum I_{\text{Bi(Pb,La)2212}}}{\sum I_{\text{Bi(Pb,La)2223}} + \sum I_{\text{Bi(Pb,La)2212}} + \sum I_{\text{Ca}_2\text{PbO}_4}};$$

and

$$V_{\text{Ca}_2\text{PbO}_4}\% = \frac{\sum I_{\text{Ca}_2\text{PbO}_4}}{\sum I_{\text{Bi(Pb,La)2223}} + \sum I_{\text{Bi(Pb,La)2212}} + \sum I_{\text{Ca}_2\text{PbO}_4}}. \quad (1)$$

respectively. Where the values of  $I$  represent the maximum intensity of each phase. We have used the peaks located in the interval  $2\theta = 15-50^\circ$ , in accordance in the work of Taghipour et al [20]. The results of the volume ratios are grouped in Table 2.

**Table 2.** Relative volume fractions of the Bi (Pb, La)-2223, Bi (Pb, La)-2212 and  $\text{Ca}_2\text{PO}_4$  phases for all samples

| Content y of La | Volume fraction of phase formed, % |                        |                                |
|-----------------|------------------------------------|------------------------|--------------------------------|
|                 | Bi (Pb, La)-2223 phase             | Bi (Pb, La)-2212 phase | $\text{Ca}_2\text{PO}_4$ phase |
| 0               | 82.434                             | 14.958                 | 2.607                          |
| 0.01            | 78.699                             | 17.893                 | 3.407                          |
| 0.02            | 73.994                             | 22.453                 | 3.553                          |
| 0.03            | 62.192                             | 31.837                 | 5.970                          |
| 0.04            | 65.306                             | 29.623                 | 5.065                          |

The quantitative results indicate that La substitution modifies the phase balance of the samples, with a change in the relative amount of the superconducting Bi (Pb, La)-2223 and Bi (Pb, La)-2212 phases. Overall, we can conclude that the dominant phase is Bi (Pb, La)-2223 and that the replacement of the calcium sites with lanthanum promotes the formation of the Bi (Pb, La)-2221 phase and inhibits that of the Bi (Pb, La)-2223 phase, these results are similar compared to those obtained by Rodrigues et al. [21], the authors found that the the substitution of La in Sr sites increase the formation of the BSCCO phases.

All of the samples' SEM images, captured at the same magnification, are displayed in Fig. 3. These micrographs clearly show the lamellar structure, which is typical of high critical temperature superconductors of Bi (Pb)-2223 grains. The creation of plate-like grains with varying forms and random orientation is visible in every photograph. The undoped sample's grains seem to be most densely packed and linked are in good agreement with reference [22]. The result for the lowest content of La ( $y = 0.01$ ) shows that the size of the grain is maximum with a mean value greater than  $5 \mu\text{m}$ . A quick and disorganized crystalline development of the grain is indicated by a greater increase in apparent porosity and a greater fall in average grain size when the concentration of Lanthanum is increased beyond 0.01.

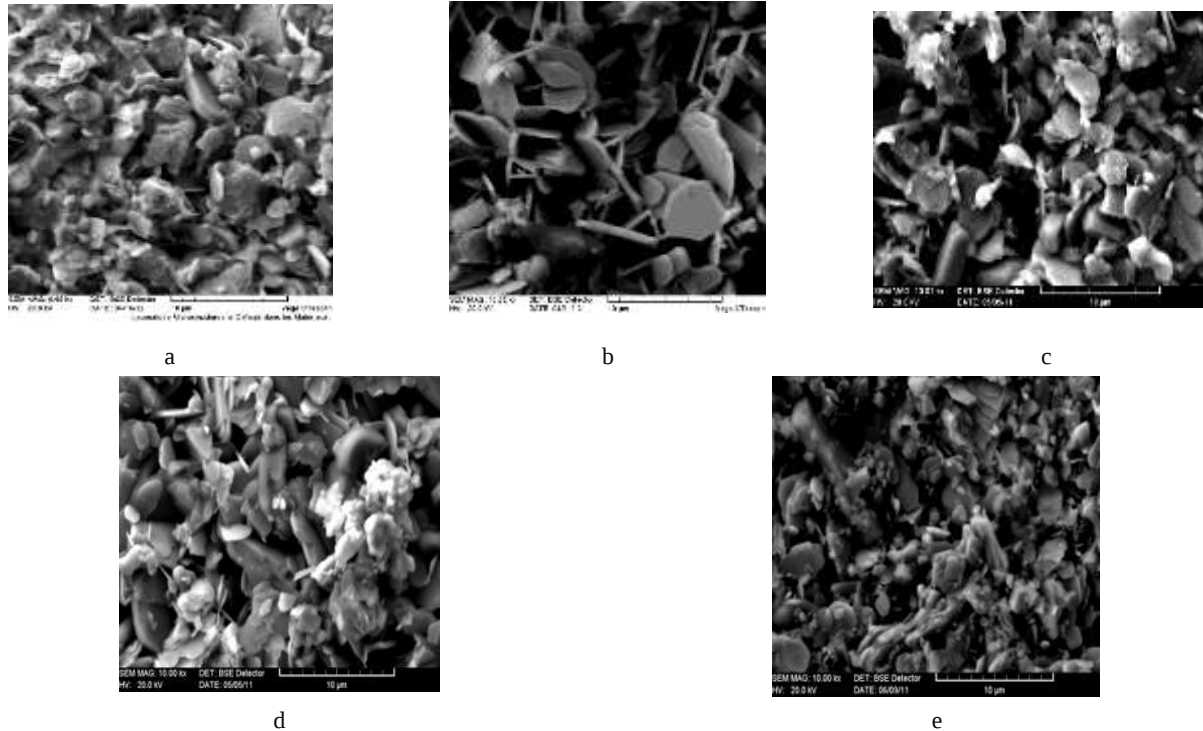
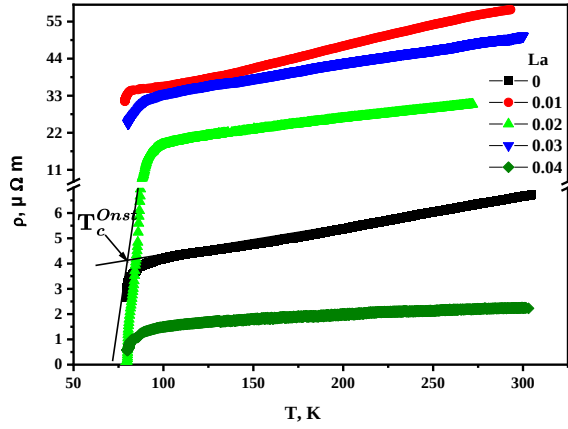
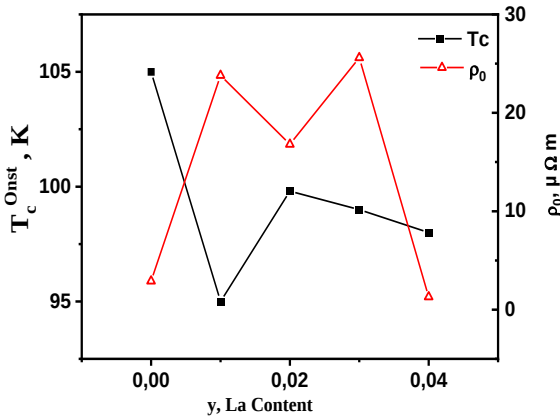
**Fig. 3.** SEM microphotographs of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+d}$  samples with the y of La content: a –  $y = 0$ ; b –  $y = 0.01$ ; c –  $y = 0.02$ ; d –  $y = 0.03$ ; e –  $y = 0.04$ .

Fig. 4 shows the variation of the resistivity  $\rho(T)$  as a function of temperature for different levels of lanthanum substitution ( $x = 0 - 0.04$ ). The critical temperature  $T_c^{Onst}$  was obtained by the intersection between the tangent drawn at the point of maximum slope of the superconducting transition and the extrapolated resistivity of the normal state.



**Fig. 4.** Resistivity as function of temperature of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+d}$ .

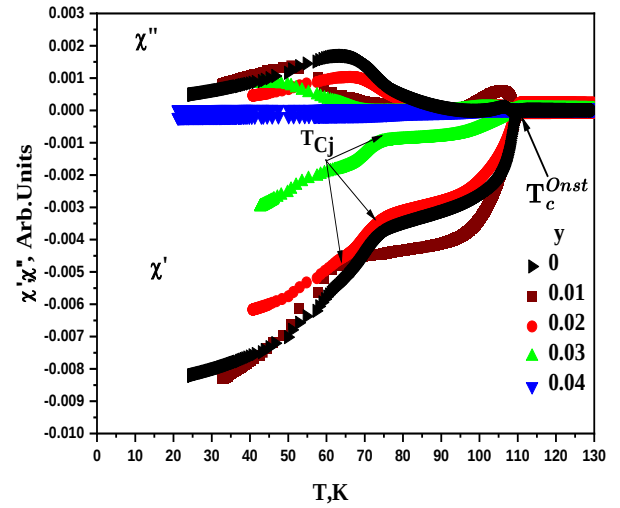
In the normal state, the resistivity decreases almost linearly with decreasing temperature, followed by a sharp drop as the material enters the superconducting transition. All samples exhibit marked metallic behavior, which decreases slightly with increasing La content. The normal-state portion of  $\rho(T)$  is reproduced by the residual resistivity  $\rho_0$ , determined by extrapolating the high-temperature  $\rho(T)$  to 0 K [23–31]. The variation of  $\rho_0$  and the critical temperature  $T_c^{Onst}$  as a function of lanthanum content is illustrated in Fig. 5.



**Fig. 5.** Critical temperature of the onset of the transition  $T_c^{Onst}$  and residual resistivity  $\rho_0$  versus the  $y$  content of La of the  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+d}$  doped samples.

The replacement of  $\text{Ca}^{+2}$  ions by  $\text{La}^{+3}$  ions leads to a reduction the number of holes in the  $\text{CuO}_2$  planes, which is accompanied by a decrease in the critical temperature  $T_c^{Onst}$ . This behavior is observed only at low La concentrations, where as for  $y = 0.04$ , the residual resistivity  $\rho_0$  becomes lower than that of the undoped sample [12]. The temperature dependence curves of the real part  $\chi'$  and the imaginary part  $\chi''$  of AC susceptibility of different La doped Bi (Pb)-2223 samples is displayed in Fig. 6. The real part for all polycrystalline samples shows clearly two transitions as the temperature is lowered below the onset of the diamagnetic

transition, which correspond to the flux removal from intra-grain and inter-grain regimes.



**Fig. 6.** AC susceptibility measurement as function of temperature of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-y}\text{La}_y\text{Cu}_3\text{O}_{10+d}$ .

The first sharp drop at the critical temperature  $T_c^{Onst}$  where the superconducting transition occurs with the generation of superconducting screening currents individual grains. Decreasing more the temperature, an intergranular regime starts at the second drop at temperature referred to  $T_{CJ}$ . Substitution of La in Ca site reduces  $T_c^{Onst}$  and increases much more the width of the transition indicating an effect on the homogeneity.

Sample of lowest content of La ( $y = 0.01$ ) has the maximum of the width of transition. SEM image of the same sample shows the formation of large angle between high sized grains, which is fatal for the grain connectivity. The imaginary part  $\chi''$  gives information about the dissipation in the sample, which is a signature of the defects. This is characterized by the position of the maximum of  $\chi''(T)$  and by its area. This latter is proportional to dissipation effects due to movement of vortices in the sample. The area of  $\chi''(T)$  decreases and shifted to lower temperature with increasing the doping level of La more than 0.01, passing by a maximum for  $y = 0$ . These results indicate increasing defects in grains and in inter grains.

## 4. CONCLUSIONS

By starting with the Lanthanum doped (Bi, Pb)-2212 precursor phase, successful preparations were made of Lanthanum doped (Bi, Pb)-2223. The results show that the incorporation of a low Lanthanum content ( $y < 0.04$ ) modifies the structural and superconducting properties of the (Bi, Pb)-2223 phase, particularly the carrier concentration, lattice parameters, orthorhombicity, and critical temperature ( $T_c$ ). Among the samples containing Lanthanum, the highest orthorhombicity is noted in the sample with the highest  $T_c$ , indicating a potential relationship between these two parameters. However, this correlation should be considered an experimental trend, not a direct cause-and-effect relationship. At low concentrations of Lanthanum, the  $c$  cell parameter begins to decrease, then subsequently increases to levels higher than those of the

undoped sample. There may be an association between the initial drop and alterations in oxygen levels and the redistribution of oxygen within the structure. Whereas any subsequent increase might be linked, at least partially, to the replacement of  $\text{La}^{3+}$  ions, whose ionic radius varies from that at the crystallographic sites substituted. With increasing Lanthanum content, a progressive decrease in  $T_c$  is observed, accompanied by an increase in structural defects. This indicates that high Lanthanum substitution levels negatively impact superconducting properties, likely via changes in carrier concentration and increases the defects in the sample. Overall, the results show that a modest Lanthanum substitution can markedly affect the structural and superconducting properties of the (Bi, Pb)2223 phase, whereas increased Lanthanum concentrations often compromise its superconducting performance.

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