

## Impact of Bi<sub>2</sub>O<sub>3</sub> addition on the normal state properties of Bi<sub>3.4</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>1.3-x</sub>RE<sub>x</sub>Cu<sub>2</sub>O<sub>y</sub> ceramics

Abdullah Aljaafari, A. Sedky\*, Ayman Al-Sawalha

Physics Department, Faculty of Science, King Faisal University, P.O.B. 400, Al-Hassa 31982, Saudi Arabia

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### ABSTRACT

The impact of Bi<sub>2</sub>O<sub>3</sub> addition on the structural, microhardness (Vickers microhardness (VHN)), resistivity and Hall coefficient measurements of Bi<sub>3.4</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>1.3-x</sub>RE<sub>x</sub>Cu<sub>2</sub>O<sub>y</sub> ceramic samples with various *x* and RE is investigated. X-ray diffraction results indicate that all the peaks belong to Bi: 2212 and 2201 phases. The moderate amount of 2212 phase exists as a major phase, while the 2201 phase as a minor. The net intensity of all peaks *I*<sub>max</sub> for 2212 phase increases with *Y* as compared to undoped sample, followed by a gradual decrease with further increase in rare earth (RE) up to La. While the vice versa is recorded for both 2201 phase and VHN. Except *Y* sample, a good link between flake-type grains is observed in the SEM photographs of the considered samples. The electrical resistivity turns from quiet metallic for undoped sample to semiconducting behavior for all substituted samples without any transition to the superconducting state. The Hall coefficient sign is found to be positive for undoped and *Y* samples and it changed from positive to negative for the other RE-substituted samples. The possible reasons for superconductivity quenching by RE substitution in this type of ceramic materials are reported.

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### 1. Introduction

Normal state properties of high *T<sub>c</sub>* superconductors have attracted significant interest in the field of superconductivity. In fact these compounds show unusual normal state properties, and it is expected that a proper understanding of their properties in the normal state may provide clues to the origin of superconductivity in this class of materials [1]. It is well known that the normal state properties of high *T<sub>c</sub>* materials could be changed with changing carrier concentration. One of the most striking results is the Hall coefficient. For instance, in the case of the high *T<sub>c</sub>* systems, the Hall coefficient *R<sub>H</sub>* is positive, and usually decreases with increasing carrier concentration [2–4]. In the under-doped region, *R<sub>H</sub>* roughly scales with the inverse of carrier concentration, which is consistent with the above picture. However, it strongly deviates from the inverse of the carrier concentration with further increase in carrier concentration, which strongly suggests that the electronic structure is altered by the hole doping. Thus, it can be believed that the appearance of superconductivity may be related to the change in the electronic structure near the Fermi surface.

BSCCO ceramic system has been characterized by a wide variety of techniques. Practical applications will require, in particular, the evaluation of its mechanical properties. Owing to their high brittleness, BSCCO ceramics exhibit very low ductility. The plasticity and strength of BSCCO can be severely affected by numerous defects such as dislocations, twins, ordering defects, microcracks and pores. Therefore, improvement of the mechanical properties of BSCCO ceramics is a major research objective [5–12]. On the other hand, extensive experimental and theoretical efforts have been devoted to understand other unusual transport properties of ceramic materials, like electrical resistivity, Hall effect, Specific heat and thermoelectric power [13–17]. Therefore, some studies based on resistivity measurements can provide valuable information about the superconductivity in these ceramic materials.

It has been reported that the mismatch of the valence state (Bi<sup>3+</sup>/Pb<sup>2+</sup>) produces a change in the electronic density of BSCCO system and thereby the superconducting properties could be improved. The reduction in the valence state of Bi<sup>3+</sup> to Bi<sup>3-δ</sup> gives rise to an electronic conduction in Bi–O planes, while the oxidation of Cu<sup>2+</sup> to Cu<sup>2+δ</sup> gives rise to the hole conduction in Cu–O<sub>2</sub> planes [18,19]. On the other hand, the substitution of Ca by RE elements in Bi:2212 systems leads to a structural stability and helps in understanding the nature and variation of charge carriers in these materials [20–31]. Most of studies have concluded that even though RE substitution improves the structural stability of Bi:2212 systems, the superconducting properties show a degrading tendency as the dopant content increases.

\* Corresponding author. Permanent address: Physics Department, Faculty of Science, Assiut University, Assiut, Egypt.

E-mail address: [sedky1960@yahoo.com](mailto:sedky1960@yahoo.com) (A. Sedky).

The present work is carried out to determine the impact of  $\text{Bi}_2\text{O}_3$  addition on the electrical resistivity, microhardness and Hall coefficient measurements of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  ceramic samples with various  $x$  and RE ( $x = 0.00$  and  $0.70$  and RE = Y, Gd, Nd and La). Furthermore, the phase purity and microstructure characterization of the samples are tested by using X-ray diffraction (XRD) and scanning electron microscope (SEM) techniques.

## 2. Experimental detail

$\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  samples with various  $x$  and RE ( $x = 0.00$  and  $0.70$  and RE = Y, Gd, Nd and La) are prepared by solid-state reaction method. The ingredients of  $\text{Bi}_2\text{O}_3$ , PbO, SrO,  $\text{RE}_2\text{O}_3$ ,  $\text{CaCO}_3$  and CuO of 4N purity are thoroughly mixed in the required proportions and calcined at  $810^\circ\text{C}$  in air for 16 h. This exercise is repeated three times with intermediate grinding at each stage. The resulting powder is reground, mixed, pressed into pellets and sintered in air at  $850^\circ\text{C}$  for 30 h. Finally, the pellets are slowly cooled to room temperature. The phase purity of the samples is examined by using X-ray diffractometer with  $\text{Cu-K}\alpha$  radiation. Microstructure characterization of the samples is performed by SEM. The microhardness of the samples is determined using an MH-6 digital microhardness tester (0.098–9.8 N). The Vickers microhardness (VHN) is estimated according to the following equation:  $\text{VHN} = 0.1891(P/d^2)$ , where  $P$  is the applied load and  $d$  is the diagonal length of indenter impression. The electrical resistivity of the samples is measured by the standard four-probe technique in closed cycle refrigerator [cryomech compressor package with cryostat Model 810-1812212, USA] within the range of 10–300 K. High-quality silver paint is used for electrical contacts of the leads with the samples. Nanovoltmeter Keithley 2182, current source Keithley 6220 and temperature controller 9700 (0.001 K resolution) are used in this experiment. Finally, Hall measurements at room temperature are made using Ecopia HMS-2000/0.57 T.

## 3. Results and discussion

Fig. 1 exhibits the XRD pattern for  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  ceramic samples with various  $x$  and RE ( $x = 0.00$  and  $0.70$  and RE = Y, Gd, Nd and La). As can be seen, all the peaks belong to Bi:2212 and 2201 phases, evident from an impurity phase. It is found that a moderate amount of Bi:2212 phase already exists as a major phase, while the Bi:2201 phase being minor. The presence of 2201 peaks like (113), (115) and (109) may be due to the syntactic growth of various phases, in agreement with the reported data [32–34]. With increasing RE, some peaks are slightly shifted either to higher or lower  $2\theta$  with  $\pm 0.2^\circ$ , and also some of peaks disappeared. It is also noticed that the gradual sharpening of 2212 peaks occurs with Y, whereas 2201 phase being minor. The net intensity of the peaks  $I_{\text{max}}$  for both 2212 and 2201 phases against RE elements are listed in Table 1. It is clear that  $I_{\text{max}}$  of 2212 slightly increases with Y, followed by a gradual decrease with further increase in RE up to La sample and vice versa for 2201 phase. Moreover,  $I_{\text{max}}$  for all samples, except R = Y sample, are slightly lower than that of undoped sample. This means that the substitution by lower ionic size of RE elements (RE = Y) leads to an increase in the amount of 2212 phase in this type of ceramic system; and vice versa for higher ionic size (RE = Gd, Nd and La).

However, it has been found that the reaction of Bi–Sr–Ca–Cu–O system is carried out in three different sintered stages [35]: formation of the insulating interface at ( $680\text{--}790^\circ\text{C}$ ); formation of

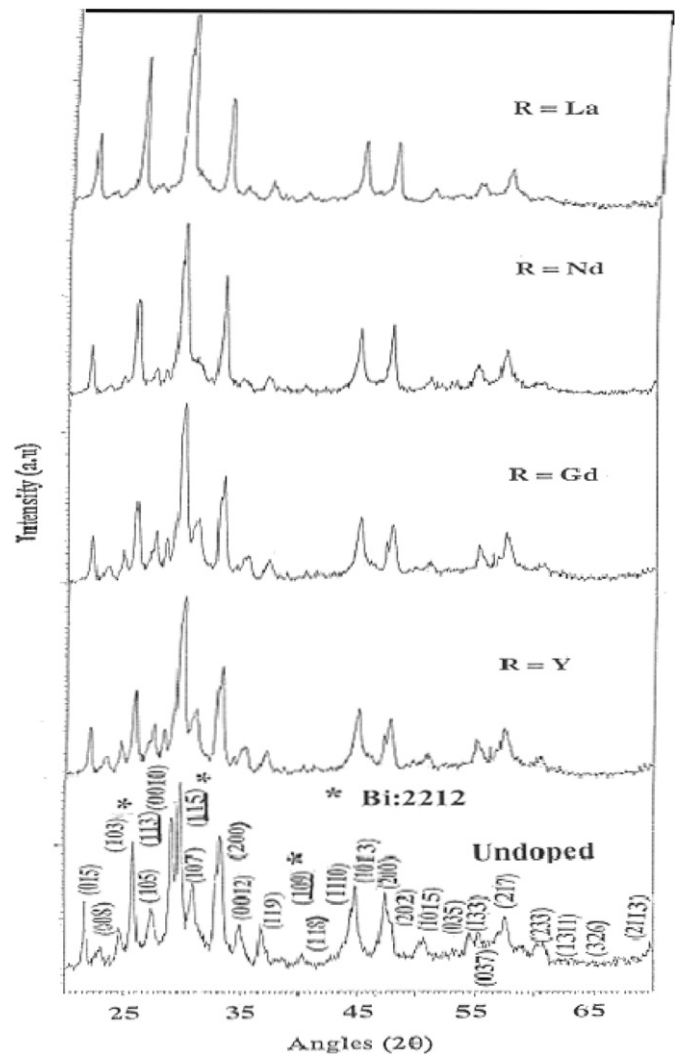


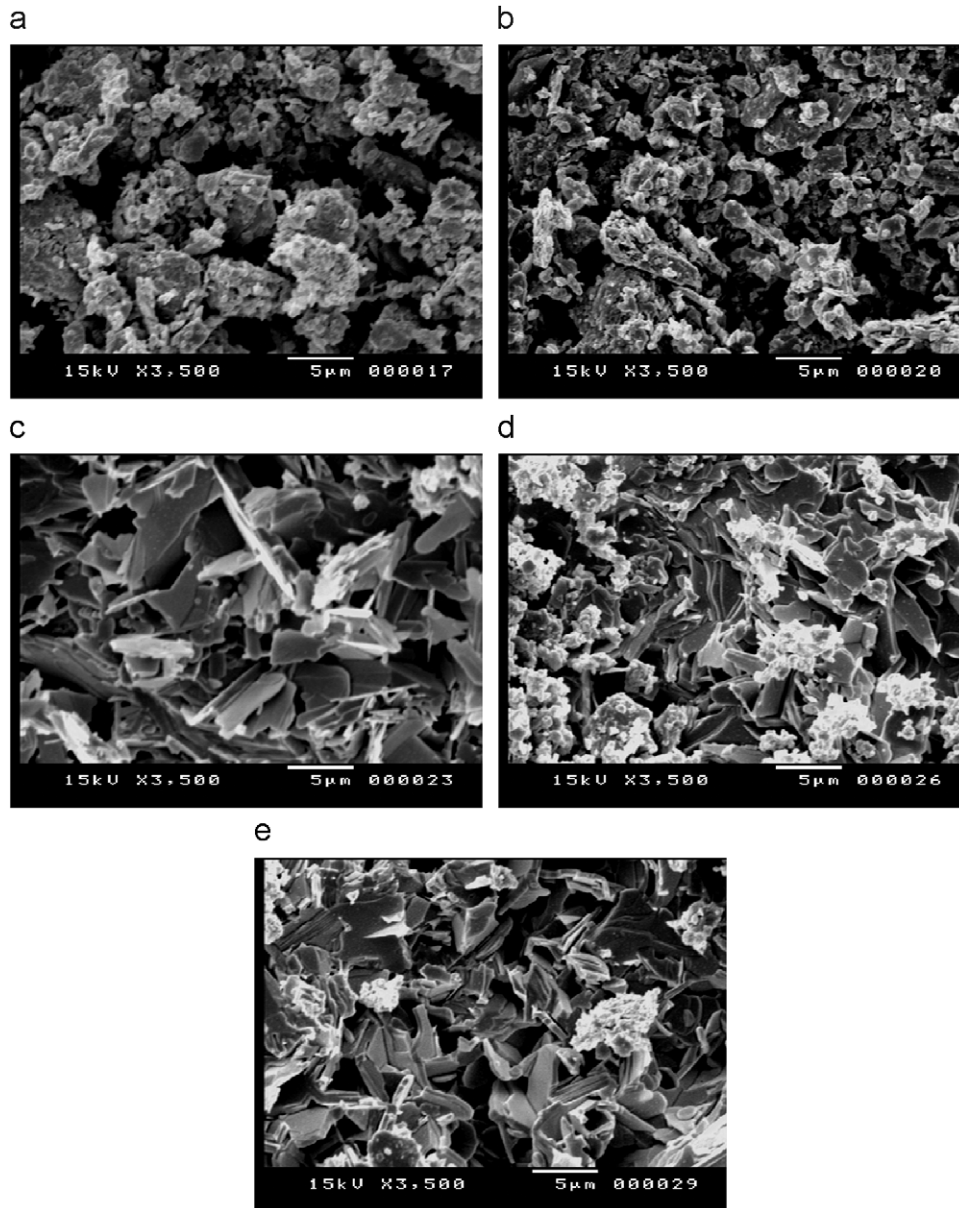
Fig. 1. XRD patterns of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  samples.

the 2212 phase at ( $790\text{--}860^\circ\text{C}$ ); and formation of the 2212 phase in the presence of the liquid phase at ( $860\text{--}970^\circ\text{C}$ ). The 2212 phase is formed by the reaction of a Bi–Sr–Ca–O interface together with CuO, SrO and Ca. The present samples are sintered at  $850^\circ\text{C}$ , which is consistent with the second stage. Therefore, the liquid phase begins to appear due to the transformation of 2212 phase to the 2201 phase, in agreement with XRD data.

The SEM micrographs for undoped and substituted  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  samples are shown in Fig. 2(a–e). In undoped sample, the number and size of flake-type grains are small, and there is a granular precipitation on the mother grains. The black regions are randomly distributed in the matrix structure, which indicate a weaker link between the grains as compared to Bi:2212 superconducting system. In Y sample, the flake-type grains are distributed randomly with few microcracks propagating along the grain boundaries, which indicate that the grains are poorly connected [36,37]. In Gd sample, the flake-shape grains appear with relatively large size, which is responsible for the low- $T_c$  phase [38]. This behavior shows a good link between the grains and also homogeneous phase distribution in the matrix structure. The small black lumps which are dispersed in the matrix may be related to some of non-superconducting phases [39]. In both Nd and La samples, the gray grains, which represent 2212 phase, are present along with a few small regions in a regular manner. The coexistence of multiphase whether

**Table 1** $I_{\max}$  of 2212 and 2202 phases, resistivity at 300 and 20K, Hall coefficient, Hall number and carrier concentration versus RE for the considered samples

RE	$I_{\max}$ 2212	$I_{\max}$ 2201	$\rho_{300}$ (m $\Omega$ cm)	$\rho_{20}$ (m $\Omega$ cm)	$R_H$ (cm <sup>3</sup> /C)	$R_H^{-1}$ (C/cm <sup>3</sup> )	Carrier concentration (cm <sup>-3</sup> )
Undoped	11,995	2327	6.18	6.31	0.0022	454.55	$2.75 \times 10^{21}$
Y	12,573	1850	100.7	139.26	0.0150	67.00	$0.41 \times 10^{21}$
Gd	11,646	1961	71	130.59	-0.0048	-208.33	–
Nd	9810	2122	53.13	105.75	-0.0110	-91.00	–
La	8448	3354	33.84	58.65	-0.0145	-69.00	–



**Fig. 2.** (a) SEM photograph of undoped sample, (b) SEM photograph of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{0.60}\text{Y}_{0.70}\text{Cu}_2\text{O}_y$  sample, (c) SEM photograph of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{0.60}\text{Gd}_{0.70}\text{Cu}_2\text{O}_y$  sample, (d) SEM photograph of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{0.60}\text{Nd}_{0.70}\text{Cu}_2\text{O}_y$  sample and (e) SEM photograph of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{0.60}\text{La}_{0.70}\text{Cu}_2\text{O}_y$  sample.

superconducting or non-superconducting are present with random distribution [40]. The presence of such phases could not be distinguished from XRD analysis, probably due to the peak overlapping with that of 2212 superconducting phase [41].

Fig. 3 shows VHN measurements plotted as a function of applied loads of 0.98, 4.9 and 9.8 N, respectively. It can be observed that at all applied loads, an approximately monotonically linear decrease in VHN with RE with a minimum at RE = Y.

After that it went up marginally as the RE substitution increased up to La sample. However, it has been reported that the substitution of Y up to 0.35 at Ca site in Bi:2212 system can substantially suppress microcracking, improve the ductility of Bi:2212 system and consequently, the microhardness (VHN) is improved [16]. In contrast, with enrichment of Y (0.55 and 0.75), the VHN is markedly reduced and this reduction is attributed to the presence of secondary phases and defects at grain boundaries

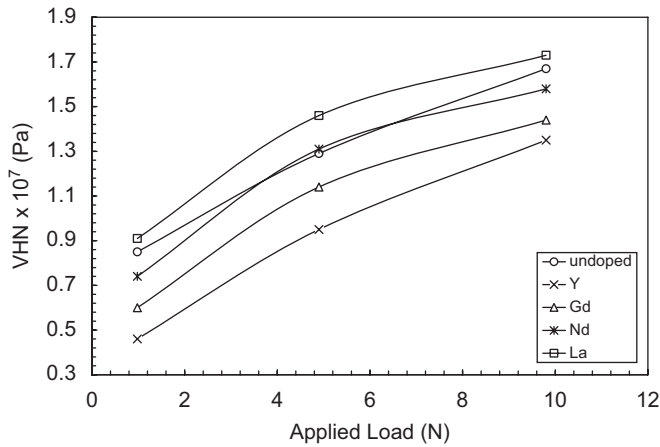


Fig. 3. The variation of microhardness with RE elements for  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  samples.

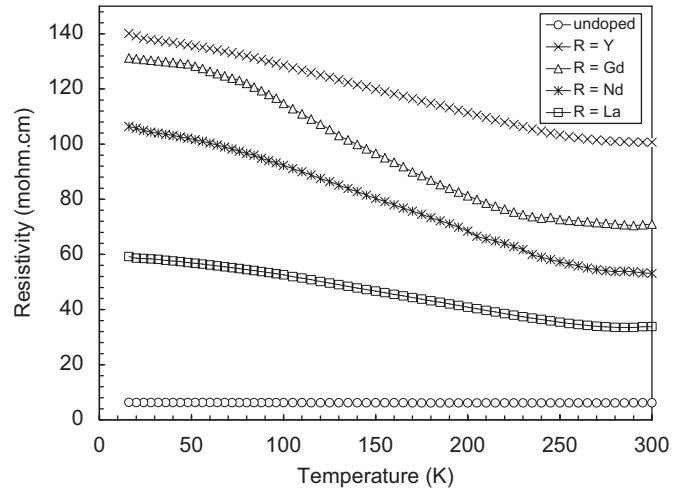


Fig. 4. Resistivity versus temperature for  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  samples.

[11]. The presence of these defects produces fine microcracks which have deleterious effect on the mechanical resistance [42]. Since the amount of Y content is 0.70 for the considered samples, the reduction of VHN for RE = Y compared with undoped sample is consistent with the previous explanation, while, the substitution of higher RE, such as Gd, Nd and La, may suppress microcracking and consequently VHN is improved. On the other hand, it can be concluded that the higher VHN values, measured at lower loads (0.98 N), represent the VHN of monocrystalline grains, while, the higher VHN values, measured at higher loads (9.8 N), usually resulted from crack patterns and crack propagation along the grain boundaries [34,43].

Fig. 4 shows the measured resistivity as a function of temperature for all considered samples. As evident from the figure, the electrical resistivity turns from metallic for pure sample to semiconducting behavior for all RE-substituted samples without any transition to the superconducting state. Perhaps, the Gd sample shows slightly higher sharp increase from metallic to semiconducting behavior as compared to the other samples, which may be due to the magnetic moment of Gd ion. Compared to pure sample, an increase in the resistivity values is observed for RE = Y sample, followed by a gradual decrease up to La sample. The different values of normal state resistivity at  $T = 300\text{ K}$  ( $\rho_{300}$ ) and resistivity at  $T = 20\text{ K}$  ( $\rho_{20}$ ) as a function of RE elements are listed in Table 1. It is clear that both  $\rho_{300}$  and  $\rho_{20}$  increase and go to maximum for RE = Y, followed by a decrease with further increase in RE up to La. Interestingly, comparing the results of resistivity with 2212 phase, one can see a good correlation between them. Now, our question is why  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  samples still remain non-superconductors. Also, why Gd-, Nd- and La-substituted samples show lower resistivity as compared to Y substituted sample. Of course, this may be due to changing of carrier concentration which will be discussed in the next section.

The Hall coefficient is measured at room temperature and at a magnetic field of 0.57T. The values of Hall coefficient for all samples are listed in Table 1. It is clear that the sign of Hall coefficient is positive only for undoped and Y samples, which indicates that the conduction is p-type for these two samples, while, the sign of Hall coefficient changed from positive to negative for Gd-, Nd- and La-substituted samples, which indicates that the conduction is n-type. The values of Hall number, listed in Table 1, are  $454\text{ C/cm}^3$  for pure sample and goes down to  $67\text{ C/cm}^3$  for Y-substituted sample. These values are slightly lower than the reported for pure Bi:2212 system ( $600\text{ C/cm}^3$ ) [44–46]. Also, the

carrier concentration, listed in Table 1, are found to be  $2.75 \times 10^{21}\text{ cm}^{-3}$  for undoped sample and decreased to  $0.41 \times 10^{21}\text{ cm}^{-3}$  for Y sample. These values are slightly lower than that reported for Bi:2212 system ( $3.0 \times 10^{21}\text{ cm}^{-3}$ ) [46,47], while, the values of both Hall number and carrier concentration for Y sample are in good agreement with those reported for Y-doped Bi:2212 system [46].

However, it has been found that the substitution of  $\text{La}^{3+}$  at  $\text{Sr}^{2+}$  site in La:214 superconductor introduces holes and the sign of Hall coefficient  $R_H$  could be changed from positive to negative above 0.32 of La content [48,49]. Further, for  $x \geq 0.15$ , the carrier concentration rises significantly above the chemical concentration value ( $n_{\text{chem.}} = 2x/V$ ), where  $x$  is the doping content and  $V$  is the volume of unit cell. Also, the sign of  $R_H$  has been found to be negative in bismuth superconductor at low magnetic field [50]. The authors attributed this unusual behavior to the curvature of the Fermi surface of ceramic systems which strongly changes with increasing doping content above 0.30.

In such a case the conduction band cannot be regarded as parabolic and the standard equation ( $n_H = 1/eR_H$ ) is found to be no longer valid to calculate carrier concentration, and other formulation is convenient in such case. Perhaps, this work is not the aim of the present study. But we can successfully reflect the present behavior of structural, mechanical and electrical properties to the curvature of the Fermi surface produced by RE substitution in these types of ceramic materials. It is found that the curvature of the Fermi surface by RE elements occurs only with higher ionic radii of RE elements such as Gd (1.06 Å), Nd (1.12 Å) and La (1.18 Å). Anyhow, the change of  $c$ -axis in the RE-substituted Bi:2212 system may be associated with the oxygen excess arising from the replacement of two CaO by one  $\text{RE}_2\text{O}_3$  molecule in the sample. It has been reported that this oxygen excess is taken up by the BiO double planes, causing a tighter binding and hence reducing the  $c$ -parameter, and consequently superconductivity is suppressed [51–53]. On the other hand, the thermal expansion measurements [54,55] based on BSCCO systems have revealed that the sample with more oxygen excess may be empty from the double Bi–O layers and therefore more positive charges will be transferred to the Cu–O layer. As a result, the carrier concentrations will be increased and the system becomes less anisotropic. In the present case, the addition of  $\text{Bi}_2\text{O}_3$  probably modifies the double Bi–O layers and the carrier concentration will be decreased, and consequently the system becomes more anisotropic. This is in good agreement with the sign of thermoelectric power  $S$  which is changed from positive to negative in the region close to room temperature ( $T \geq 250\text{ K}$ ) for



Y-substituted Bi:2212 samples [16]. The change of  $S$  sign from positive to negative indicates that the electron carriers dominate the hole carriers, and the conduction becomes n-type. Now, why we could not obtain superconductivity in pure and Y samples? We believe that the formation of Bi–O double layers suppressed the reaction, localized the hole carriers and quenched superconductivity in these types of material [35]. Perhaps the present study gives us the hope for the superconductivity in La-substituted BSCCO system.

#### 4. Conclusion

Structural and transport properties of  $\text{Bi}_{3.4}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{1.3-x}\text{RE}_x\text{Cu}_2\text{O}_y$  ceramic samples are investigated. We have shown that the XRD peaks belong to Bi:2212 and 2201 phases. Compared to undoped sample,  $I_{\text{max}}$  of 2212 phase is increased with Y, followed by a gradual decrease with further increase in RE up to La; while the vice versa for both 2201 phase and microhardness. A good link between flake-type grains is observed in the SEM photographs of RE-substituted samples. Except undoped sample which show quiet metallic, all substituted samples show semiconducting behavior and the transition to superconducting state could not be observed. Although, the sign of Hall coefficient is positive for undoped and Y samples, it changed from positive to negative for the other RE-substituted samples. The localization of hole carriers and the curvature of the Fermi surface, which are produced by both  $\text{Bi}_2\text{O}_3$  addition and RE substitution, are found to be the main reasons for the absence of superconductivity in this type of samples. Perhaps the present study gives us the hope for the superconductivity in La-substituted sample.

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