

# Synthesis of new Pb-based layered cuprates in (Pb,S)(Sr,La)CuO<sub>z</sub> compounds

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## Abstract

The effect of sulfate substitution on the formation of (Pb,S)-1201 type phase was investigated. Polycrystalline samples with nominal compositions of  $(\text{Pb}_{0.5}\text{B}_{0.5-x}\text{S}_x)(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$ , ( $x = 0 - 0.5$ ,  $y = 0.7 - 1.0$ ) and  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  ( $y = 0.5 - 1.0$ ) were prepared by using a solid-state reaction method. The samples were characterized by powder X-ray diffraction (XRD) and resistivity measurements. XRD data revealed that almost-single (Pb,S)-1201 phase samples could be obtained for  $x = 0.5$  and  $y = 0.9-1.0$ , judging from the similar results of the XRD patterns between the (Pb,S)-1201 and (Pb,B)-1201 phases. Each of the samples has a crystal structure with tetragonal symmetry. The sample with  $x = 0.5$  and  $y = 0.9$  is found to show an onset of resistivity dropping at over 23 K and zero resistivity at 12 K.

**Keywords:** Pb-based 1201 cuprate, superconductivity, sulfate doping,  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$

## 1. INTRODUCTION

Soon after  $(\text{Pb}_2\text{Cu})\text{Sr}_2(\text{Ca},\text{Y})\text{Cu}_2\text{O}_z$  (Pb-3212) superconductors were isolated by Cava et al.[1], the synthesis of new Pb-based superconducting layered cuprates of  $(\text{Pb}_{0.5}\text{Sr}_{0.5})\text{Sr}_2(\text{Ca}_{0.5}\text{Y}_{0.5})\text{Cu}_2\text{O}_z$  ((Pb,Sr)-1212) [2] and  $(\text{Pb}_{0.5}\text{Cu}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  ((Pb,Cu)-1201) [3,4] was reported. Thereafter, extensive efforts were devoted to the study of this kind of material and several new superconducting lead-based 1212 cuprates  $(\text{Pb},\text{M})\text{Sr}_2(\text{Ca}_{1-y}\text{R}_y)\text{Cu}_2\text{O}_z$  ( $\text{R} = \text{rare-earth element}$ ,  $\text{M} = \text{Cu}$  [5,6],  $\text{Ca}$  [7],  $\text{Mg}$  [8],  $\text{Cd}$  [9],  $\text{V}$  [10],  $\text{Ti}$  [11],  $\text{Sn}$  [12]) and  $(\text{Pb},\text{M})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  ( $\text{M} = \text{Cd}$  [13],  $\text{Zn}$  [13],  $\text{B}$  [14]) were synthesized. These findings indicate a great degree of flexibility of the rock-salt-type (Pb,M)O layer in the 1212 and 1201 cuprates. In particular, the formation of (Pb,B)-1201 phase suggests that Cu sites in the monolayer of (Pb,Cu)O can be replaced by oxyanion. In fact, Slater et al. [15] reported the first study on sulfate and phosphate substitution for Cu sites in cuprates with nominal compositions of  $\text{YSr}_2\text{Cu}_{3-x}(\text{SO}_4)_x\text{O}_y$  and  $\text{YSr}_2\text{Cu}_{3-x}(\text{PO}_4)_x\text{O}_y$  ( $0.2 < x < 0.32$ ), which can be rendered superconducting. After isolation of (Pb,B)-1201 phase, (Pb,S)-1212 [16] and (Pb,S)-1222 [17] compounds have been synthesized, but the formation of (Pb,S)-1201 phase was not reported. In this work, we attempted to synthesize (Pb,S)-1201 phase based on our recent success in isolation of sulfate-based superconducting  $\text{TiSr}_4\text{Cu}_2\text{O}_z(\text{SO}_4)$  compounds [18 - 20].

## 2. EXPERIMENTS

Polycrystalline samples having the nominal compositions of  $(\text{Pb}_{0.5}\text{B}_{0.5-x}\text{S}_x)(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$ , ( $x = 0 - 0.5$ ,  $y = 0.7 - 1.0$ ) and  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  ( $y = 0.5 - 1.0$ ) were prepared by using a solid-state reaction from starting powders of PbO, B<sub>2</sub>O<sub>3</sub>, CuSO<sub>4</sub>, SrO<sub>2</sub>, La<sub>2</sub>O<sub>3</sub> and CuO. The mixtures were thoroughly ground and cold-pressed into disk-shaped pellets. The pellets were then wrapped in a silver foil to reduce possible volatilization of Pb. The wrapped materials were heated at 895 °C for 1 h – 2 h in air and then cooled to a temperature below 200 °C in a conventional tube furnace. Some of the samples were quenched into liquid nitrogen after heating at 895 °C. X-ray diffraction (XRD) measurements to determine the phase purity and lattice parameters of the samples were performed by using a powder diffractometer (X'pert-pro MPD) with K $\alpha$  radiation at room temperature. Resistivity measurements were made on rectangular specimens cut from sintered pellets, by using a conventional four-probe technique. The measuring current was 10 mA.

## 3. RESULTS AND DISCUSSION

The XRD patterns for  $(\text{Pb}_{0.5}\text{B}_{0.5-x}\text{S}_{0.5})(\text{Sr}_{1.2}\text{La}_{0.8})\text{CuO}_z$  samples with  $x = 0, 0.25$ , and  $0.5$  are shown in Fig. 1. From the XRD patterns, one can notice that the diffraction pattern for the  $x = 0.25$  sample is nearly identical to that for the  $x = 0$  sample. A similar feature is observed for the  $x = 0.5$  sample except for slight increases in peak intensity, which are denoted by asterisks in Fig. 1. The peaks denoted by asterisks are due to impurity phases of Pb-Sr-Cu-O solid

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solution [21] which are typically observed in Pb-1201[13] systems. The main diffraction peaks except for those of the impurity phase could be indexed on the basis of a tetragonal unit cell as denoted in Fig. 1

The tetragonal cell parameters determined by the least squares fitting method were  $a = 3.791(6)$  Å and  $c = 8.657(12)$  for the  $x = 0$  sample and  $a = 3.789(3)$  Å and  $c = 8.669(6)$  Å for the  $x = 0.5$  sample. These values are comparable to the reported values of  $a = 3.779$  Å and  $c = 8.654$  Å for the (Pb<sub>0.5</sub>B<sub>0.5</sub>)(SrLa)CuO<sub>z</sub> compound [14]. These results clearly indicate the formation of (Pb, S)-1201 phase, judging from the similarity of the diffraction patterns of the samples shown in Fig. 1. These findings also contrast with the report that the Pb-1201 phase does not form for  $M = P$  or  $S$  in (Pb<sub>1-x</sub>M<sub>x</sub>)(Sr<sub>2-y</sub>La<sub>y</sub>)CuO<sub>z</sub> systems [14]. Fig. 2 shows the temperature dependences of the electrical resistivity for the (Pb<sub>0.5</sub>B<sub>0.5-x</sub>S<sub>x</sub>)(Sr<sub>1.2</sub>La<sub>0.8</sub>)CuO<sub>z</sub> samples. The sample with  $x = 0$  exhibits a superconductivity onset temperature  $T_c$  (onset) = 17 K and zero-resistivity temperature  $T_c$  (zero) = 8 K. For the S-doped samples, both  $T_c$  (onset) and  $T_c$  (zero) values are enhanced as compared to the S-free  $x = 0$  sample and the  $x = 0.5$  sample shows  $T_c$  (onset) = 22 K and  $T_c$  (zero) = 13 K.

Fig. 3 shows the electrical resistivity data for (Pb<sub>0.5</sub>S<sub>0.5</sub>)(Sr<sub>2-y</sub>La<sub>y</sub>)CuO<sub>z</sub> ( $y = 0.5 - 1.0$ ) samples. The  $y = 1.0$  sample (a) shows a  $T_c$  (onset) at 22 K but does not reach zero resistivity on lowering the temperature to about 10 K. However, when La-content is decreased below  $y = 0.9$ , the  $T_c$  (zero) value tends to increase until  $y = 0.7$ . A further decrease in  $y$  below  $y = 0.7$  results in a decrease in the  $T_c$  (zero) values and a prominent increase in normal-state resistivity as shown in the inset of Fig. 3. Although the  $T_c$  (zero) value changes with the La-doping content  $y$ , one can notice that the  $T_c$  (onset) values hardly changes, that is, the  $T_c$  (onset) values are 22 K, 23 K, 22 K, 23 K, 21 K and 23 K for  $y = 1.0, 0.9, 0.8, 0.7, 0.6,$  and  $0.5$ , respectively. Fig. 4 shows the XRD patterns for (Pb<sub>0.5</sub>S<sub>0.5</sub>)(Sr<sub>2-y</sub>La<sub>y</sub>)CuO<sub>z</sub> ( $y = 0.5 - 1.0$ ) samples. The main diffraction pattern for the  $y = 1.0$  sample shows the diffraction pattern of the pure Pb-1201 phase. The intensity of the impurity peaks marked by asterisks increased as the La-content decreased from  $y = 1.0$  and a significant

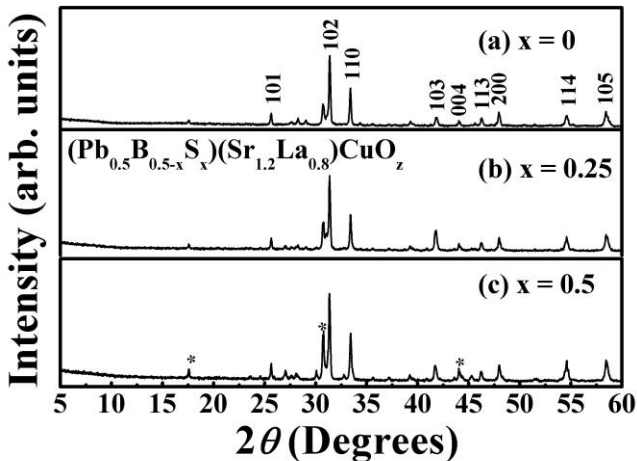


Fig. 1. Powder XRD patterns for (Pb<sub>0.5</sub>B<sub>0.5-x</sub>S<sub>x</sub>)(Sr<sub>1.2</sub>La<sub>0.8</sub>)CuO<sub>z</sub> samples.

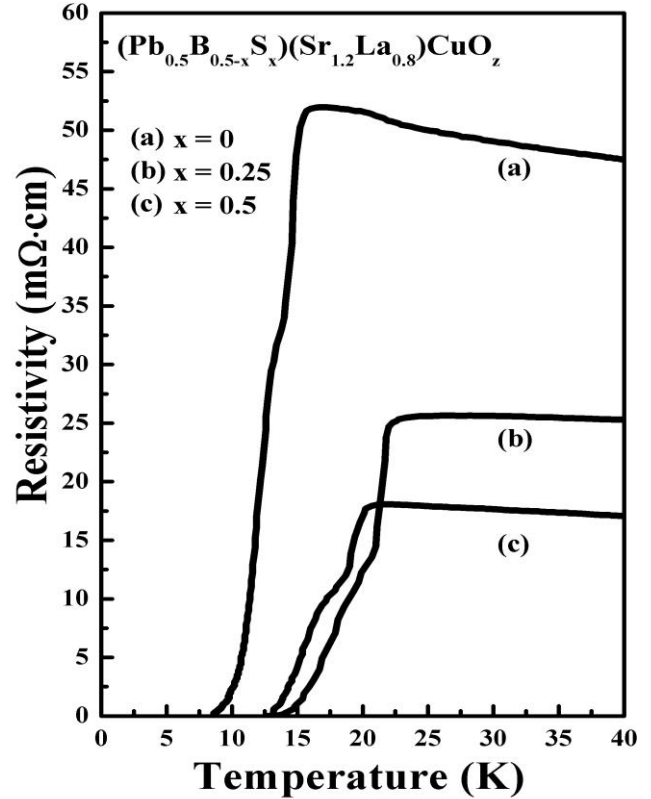


Fig. 2. Temperature dependences of the electrical resistivity for (Pb<sub>0.5</sub>B<sub>0.5-x</sub>S<sub>x</sub>)(Sr<sub>1.2</sub>La<sub>0.8</sub>)CuO<sub>z</sub> samples.

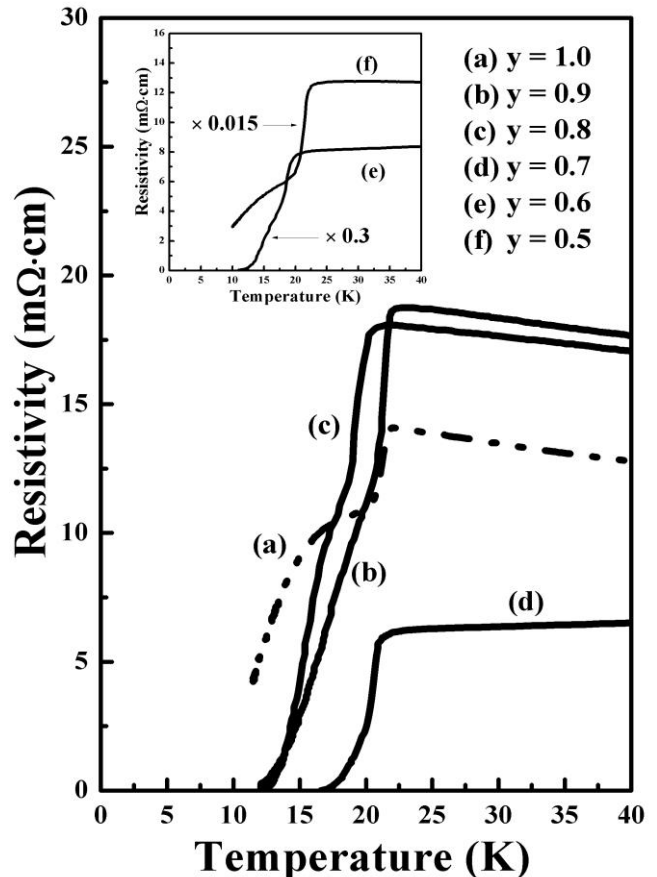


Fig. 3. Temperature dependences of the electrical resistivity for (Pb<sub>0.5</sub>S<sub>0.5</sub>)(Sr<sub>2-y</sub>La<sub>y</sub>)CuO<sub>z</sub> samples.

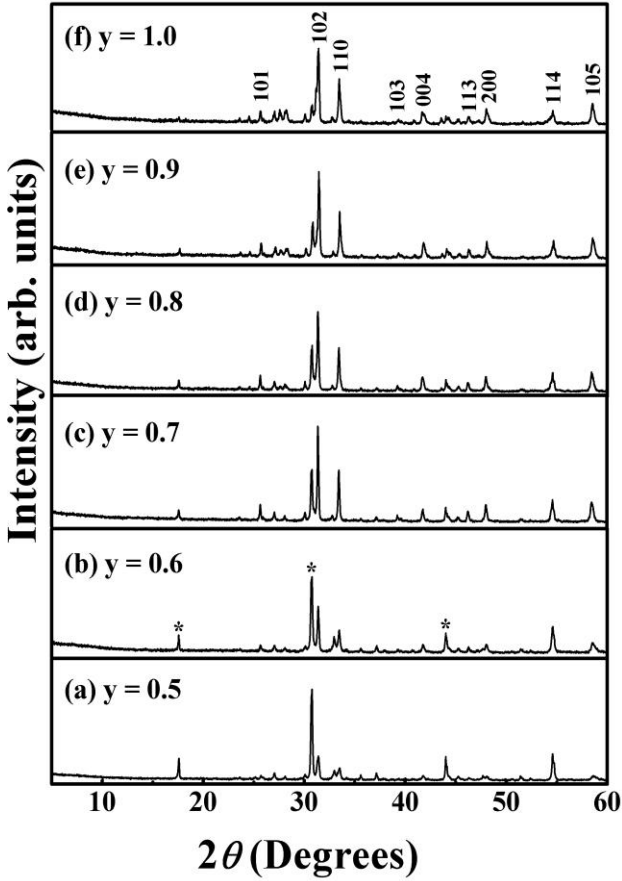


Fig. 4. Powder XRD patterns for  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  samples.

increase in impurity content was observed for the samples with  $y < 0.7$ . Although it is not shown, a similar increase of the same impurity phase with decreasing La doping content was also observed for the  $(\text{Pb}_{0.5}\text{B}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  ( $y = 0.7 - 1.0$ ) system. The cell parameters determined from the diffraction peaks for the  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  samples were  $a = 3.786(2) \text{ \AA}$  and  $c = 8.665(4) \text{ \AA}$  for the  $y = 1.0$  sample and  $a = 3.787(4) \text{ \AA}$  and  $c = 8.656(8) \text{ \AA}$  for the  $y = 0.7$  sample. Considering the difference in ion size [22] between La ( $1.16 \text{ \AA}$  for CN = 8) and Sr ( $1.26 \text{ \AA}$  for CN = 8), the small change in the lattice parameters and unit cell volume seems to suggest that the La doping content does not vary according to the nominal compositions. For polycrystalline high- $T_c$  cuprate superconductors, the hole concentration per  $\text{CuO}_2$  plane plays a very important role and  $T_c$  (onset) is considered to be determined mainly by the carrier concentration of the grains. However, the zero resistivity transition depends not only on the carrier concentration but also on the grain connectivity [23]. The grain connectivity can be greatly affected by the formation of impurity phase. Therefore, the prominent increase in the normal-state resistivity and the decrease in  $T_c$  (zero) values for the samples with  $y < 0.7$  are explained by the notable increase in impurity content. On the other hand, if the La content in Sr sites is changed,  $T_c$  (onset) is expected to change as well due to the valence difference between  $\text{La}^{+3}$  and  $\text{Sr}^{+2}$ . Thus, despite the change in nominal content of La in the

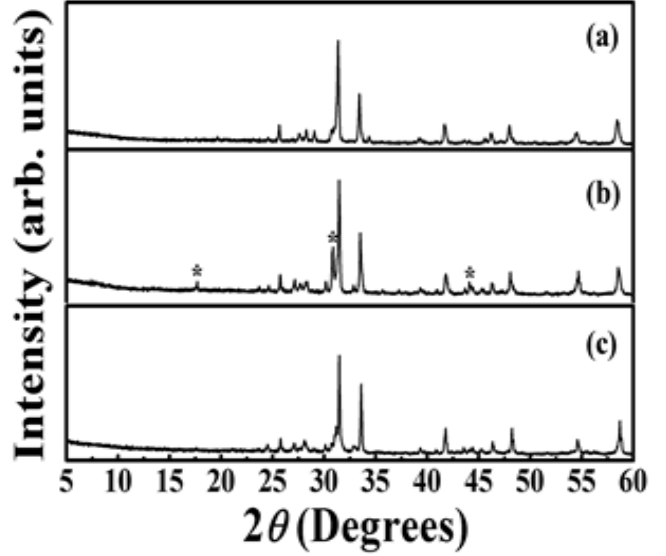


Fig. 5. Powder XRD patterns for (a)  $(\text{Pb}_{0.5}\text{B}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$ , (b)  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$ , and (c) quenched  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$  samples.

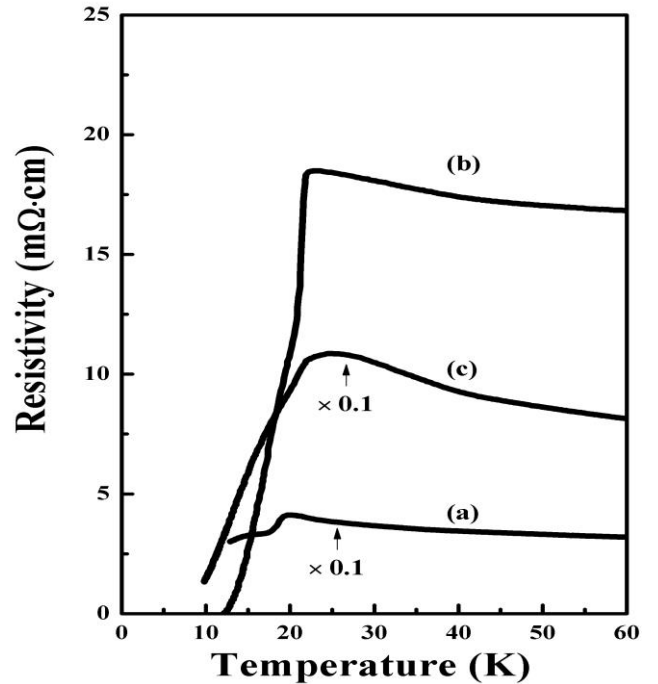


Fig. 6. Temperature dependences of the electrical resistivity for (a)  $(\text{Pb}_{0.5}\text{B}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$ , (b)  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$ , and (c) quenched  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$  samples.

composition, the nearly constant value of the  $T_c$  (onset) suggests that the soluble La content in the  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  system is very limited. Combined with the increase in the impurity content with the decrease in La doping content below  $y = 0.9$  as shown in Fig. 4, the soluble La doping content is considered to be  $y = 0.9-1.0$ .

Fig. 5 displays the XRD patterns for (a)  $(\text{Pb}_{0.5}\text{B}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$ , (b)  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$ , and (c) quenched  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{1.1}\text{La}_{0.9})\text{CuO}_z$  samples. The

quenched sample was prepared by heating at 895 °C for 1 h in air and subsequent quenching in liquid nitrogen. Comparing the XRD patterns of the (b) and (c) samples, the impurity peak is significantly reduced after the quenching operation. This result suggests that nearly phase-pure (Pb,S)-1201 phase can be formed under appropriate synthesis conditions. Fig. 6 shows the resistivity data for the samples shown in Fig. 5. Sample (a) shows  $T_c$  (onset) at 20 K but does not reach zero resistivity on lowering the temperature to about 10 K. In contrast, sample (b) exhibits a  $T_c$  (onset) at 23 K and  $T_c$  (zero) at 12 K. The broad transition in the resistivity curve for sample (c) is considered to be caused by inhomogeneous distribution of the hole concentration in the sample. Although we have not optimized the sample preparation conditions for sample (c) at this stage, it is expected that  $T_c$  (zero) could be reached near  $T_c$  (onset) if we could optimize the preparation conditions including the sintering temperature, time and atmospheres.

#### 4. CONCLUSION

Based on the recent successful synthesis of sulfate-based  $\text{TiSr}_4\text{Cu}_2\text{O}_z(\text{SO}_4)$  compounds, we addressed sulfate substitution for M sites of the (Pb,M)O mono-layers in the crystal structure of the Pb-1201 compound  $(\text{Pb,M})(\text{Sr,La})_2\text{CuO}_z$ . Polycrystalline samples with nominal compositions of  $(\text{Pb}_{0.5}\text{B}_{0.5-x}\text{S}_x)(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$ , ( $x = 0 - 0.5$ ,  $y = 0.7 - 1.0$ ) and  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  ( $y = 0.5 - 1.0$ ) were synthesized and characterized by means of XRD and resistivity measurements. XRD data for the  $(\text{Pb}_{0.5}\text{B}_{0.5-x}\text{S}_x)(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  samples showed that almost-single 1201 phase samples were formed for  $y = 0.9 - 1.0$ , judging from the finding that the diffraction pattern for the  $(\text{Pb}_{0.5}\text{S}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  samples was nearly identical to that of the Pb-1201 phase in the  $(\text{Pb}_{0.5}\text{B}_{0.5})(\text{Sr}_{2-y}\text{La}_y)\text{CuO}_z$  system. The superconductivity transition temperature for the  $y = 0.9$  sample revealed an onset of resistivity dropping at over 23 K and zero resistivity at 12 K.

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