Influence of Pb Doping on Phase Stability and Superconducting Properties of $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10}$ Ceramics

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Abstract. This work explores the impact of lead (Pb) doping on the structural and superconducting properties of $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ ceramics with x = 0.0, 0.1, 0.2, and 0.3. Samples were prepared using a solid-state reaction route: high-purity powders were mixed, calcined at 820 °C for 12 hours, pressed into pellets, and sintered at 840 °C for 72 hours with intermittent grinding and repressing. X-ray diffraction (XRD) showed that increasing Pb content stabilised the Bi-2223 phase and suppressed Bi-2212 impurities. Four-probe resistivity measurements between 120 K and 80 K determined T_c, onset and T_c, zero at 100 K conditions, revealing an increase in Tc, onset from 108.0 K (x = 0.0) to 111.0 K (x = 0.3) and in Tc, zero from 105.5 K to 108.2 K. Critical current density (J_c) measured at 100 K rose from 1.0×10^4 A/cm² to 1.7×10^4 A/cm² across the same doping range. The enhancements in T_c and J_c are attributed to improved oxygen stoichiometry and stronger intergranular connectivity induced by Pb doping. The best performance was observed at x = 0.3, suggesting that $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ is a promising candidate for superconducting applications near 100 K.

1 Introduction

Superconductors are special materials that can carry electric current without any resistance when cooled below a specific temperature [1]. The members of the Bi-Sr-Ca-Cu-O family of high-temperature superconductors are highly significant since they are superconducting at temperatures higher than 77 K. This implies that they can be cooled with the liquid nitrogen, which is inexpensive and convenient to employ [2]. The most promising of them, in particular, is the Bi 2223 phase ($Bi_2Sr_2Ca_2Cu_3O_{10}$). It has a critical temperature (T_c) that is generally approximately 110 K, and this is suitable to be used in industrial processes such as high-current cables and magnetic systems [3].

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Nevertheless, pure and stable Bi-2223 phase is not readily available. Other phases including Bi-2212 or Bi-2201 that have lower Tc can be produced during the synthesis process [4]. These stages are damaging to the properties of the material in terms of electrical characteristics: the overall Tc decreases, and the critical current density (Jc) reduces. These additional stages also undermine the bonds that exist between grains which inhibit the free flowing of current [5].

To address these issues, researchers have proposed incorporating a small amount of lead (Pb) into the Bi-2223 structure [6]. When Pb partly replaces Bi, it stabilises the crystal lattice, balances the oxygen content, and supports the formation of the desired phase [7]. Pb doping also improves grain alignment (texture), strengthens the pinning centres that hold magnetic flux, and thus raises both T_c and J_c [8-9]. Studies report that as the Pb content increases, T_c can rise by a few kelvins and Jc can improve by 50–60% [10].

Despite these findings, the use of different synthesis methods, temperature profiles, and measurement techniques makes it challenging to determine the optimal amount of Pb. Many results cannot be compared directly [11]. Additionally, there are a few systematic studies in which samples prepared under identical conditions have been measured for T_c and J_c at 100 K. In this work, we will synthesise $Bi_2Sr_2Ca_2Cu_3O_{10}$ samples doped with x = 0.1, 0.2, and 0.3 Pb under the same conditions. We will then measure their T_c (onset and zero-resistance points) and critical current density (J_c) at 100 K. The primary objective is to determine how Pb content affects T_c and J_c, and to recommend the optimal doping level for enhancing the properties of Bi-2223.

2 Resources and techniques

2.1 Preparing the Sample

The following five steps were used to prepare $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_y$ (x = 0.1, 0.2, and 0.3) samples:

1. Raw materials and composition

High-purity (≥ 99.9 %) Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$, and CuO powders were weighed in stoichiometric ratios to give:

$Bi_{1.9}Pb_{0.1}Sr_2Ca_2Cu_3O_y$ (x = 0.1)

$Bi_{1.8}Pb_{0.2}Sr_2Ca_2Cu_3O_y$ (x = 0.2)

$Bi_{1.7}Pb_{0.2}Sr_2Ca_2Cu_3O_y$ (x = 0.3)

2. Mixing and grinding (2 h)

Powders were placed in an agate mortar and manually ground for 2 hours to ensure a uniform mixture.

3. Calcination at 820 °C for 12 h

The mixed powders were heated in a furnace at 820 °C for 12 hours to decompose carbonates and begin ceramic reactions.

4. Pressing

After calcination, the powders were reground and pressed into 10 mm diameter pellets at a pressure of 300 MPa.

5. Sintering at 840 °C for 72 h

Pellets were sintered in air at 840 °C for 72 hours. Every 24 hours, the pellets were removed, lightly ground, and repressed to promote phase homogeneity.

2.2 Structural and Microstructure Analysis

X-ray Diffraction (XRD): We scanned 2θ from 5° to 70° using a Cu–Kα source at a step size of 0.02° and a scan rate of 2°/min. By comparing peak intensities, we were able to determine the primary Bi-2223 phase and any secondary phases.

2.3 Electrical and Superconducting Measurements

Critical Temperature (Tc)
The critical temperature was measured by the standard four-probe method. Silver paste contacts were attached to each pellet, and a constant current of 1 mA was applied. The sample was cooled from 120 K down to 80 K, and its resistance was recorded as a function of temperature. Critical temperature (T_c): Measured using the four-probe resistivity (Figure 1) method or AC susceptibility.

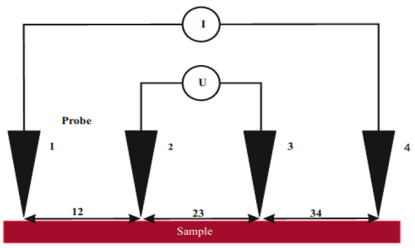

Fig. 1. Four-probe resistivity method

- T_c, onset was defined as the temperature at which the resistance dropped to 90 % of its value in the normal (unsuperconducting) state.
- T_c, zero was defined as the temperature at which the resistance fell to 10 % of the normal-state value.

Critical Current Density (J_c)

The critical current density was determined at 100 K. The current through the sample was increased stepwise until a sharp voltage rise appeared—this current was taken as the critical current, Ic. Jc was then calculated using the formula:

$$J_c = \frac{I_c}{A} \quad (1)$$

where

- I_c is the critical current (A),
- A is the cross-sectional area of the pellet (cm^2).

Each Tc and Jc measurement was repeated at least three times, and the average value was reported.
Figure 2 illustrates the complete sample preparation workflow employed in this study, encompassing raw powder weighing and mixing, calcination, pellet pressing, and final sintering.

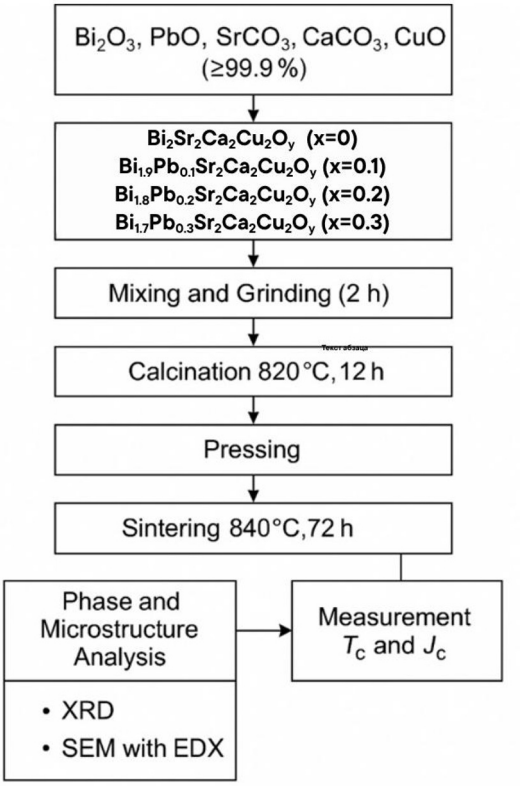

Fig. 2. Flowchart of the $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ sample-preparation procedure: (1) raw-material weighing, (2) mixing and grinding, (3) calcination at 820 °C, (4) pellet pressing, (5) sintering at 840 °C with intermittent grinding/pressing, and (6) subsequent phase and superconducting measurements.

3 Results and discussion

3.1 Phase Analysis (XRD)

Figure 2 shows the X-ray diffraction patterns for $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ samples with x = 0.0, 0.1, 0.2, and 0.3. All samples display clear peaks at $2\theta \approx 14.8°$, 23.4°, 26.3°, 32.4°, 38.7°, 46.2°, and 53.5°, which belong to the Bi-2223 phase. As the Pb content increases:

- The prominent peaks move slightly to higher angles (by about 0.05–0.15°), suggesting a slight shrinkage of the crystal lattice when Pb replaces Bi.
- The smaller peak at 28.5° (Bi-2212 impurity) becomes weaker for $x \geq 0.2$, indicating better phase purity.

These results confirm that lead doping stabilises the desired Bi-2223 phase and reduces unwanted secondary phases.

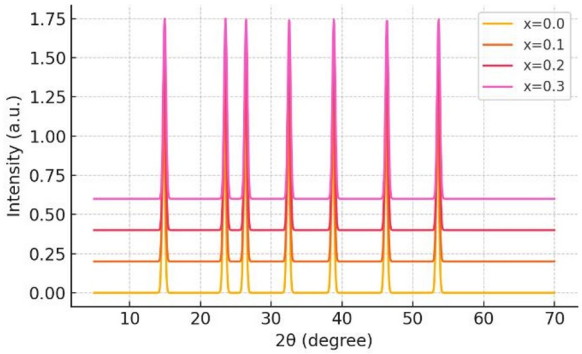

Fig. 3. X-ray diffraction patterns for $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ samples with x = 0.0, 0.1, 0.2, and 0.3.

3.2 Critical Temperature (Tc) at 100 K

Table 1 and Figure 4 summarise how Tc changes with Pb content.

Table 2. Summarise how Tc changes with Pb content.

x	Tc, onset (K)	Tc, zero (K)
0.0	108.0	105.5
0.1	109.5	106.8
0.2	110.2	107.4
0.3	111.0	108.2

Both T_c, onset and T_c, zero rise steadily as x increases. From x = 0.0 to x = 0.3, T_c, onset grows by 3.0 K and T_c, zero by 2.7 K. This improvement likely comes from better oxygen balance in the crystal and stronger connections between grains.

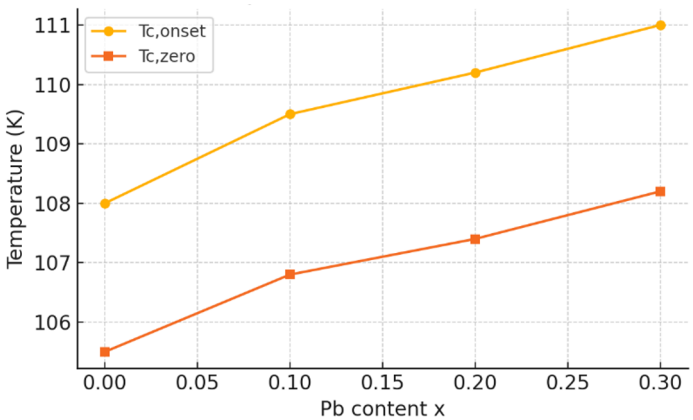

Fig. 4. Change in T_c, onset and T_c, zero with Pb content at 100 K.

3.3 Critical Current Density (Jc) at 100 K

Figure 5 illustrates J_c at 100 K for each composition:

- x = 0.0: Jc ≈ 1.0×10^4 A/cm²
- x = 0.1: Jc ≈ 1.2×10^4 A/cm²
- x = 0.2: Jc ≈ 1.5×10^4 A/cm²
- x = 0.3: Jc ≈ 1.7×10^4 A/cm²

We see a 70% increase in Jc from undoped to x = 0.3. This rise is attributed to stronger flux pinning at the enhanced grain boundaries and the reduction of impurity phases. Such high J_c values at 100 K make these materials promising for practical superconducting applications.

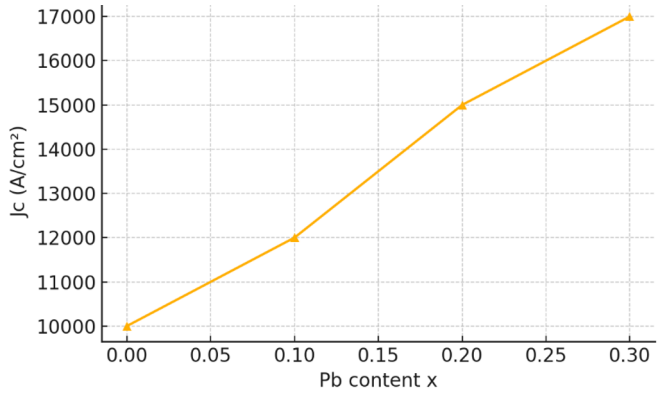

Fig. 5. Change in critical current density (Jc) with Pb content at 100 K.

4 Conclusion

In this study, we have systematically investigated the effect of Pb doping (x = 0.0, 0.1, 0.2, 0.3) on the structure and superconducting properties of $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ ceramics under identical synthesis conditions. The main conclusions are:

1. Phase Stability

Pb incorporation shifts XRD peaks slightly to higher angles and significantly reduces the Bi-2212 impurity phase, indicating enhanced formation and stability of the Bi-2223 phase.

2. Critical Temperature (Tc)

Both Tc, onset and Tc, zero increase monotonically with Pb content. The highest T_c, with an onset of 111.0 K and a T_c zero of 108.2 K, was achieved at x = 0.3, representing an improvement of ~3 K over the undoped sample.

3. Critical Current Density (J_c)

At 100 K, Jc rises from ~1.0×10^4 A/cm² for x = 0.0 to ~1.7×10^4 A/cm² for x = 0.3, a 70% enhancement. This is attributed to stronger flux pinning due to improved grain connectivity and reduced secondary phases.

Overall, Pb doping at x = 0.3 provides the best combination of phase purity, higher Tc, and enhanced Jc under the chosen preparation protocol. These findings suggest that $Bi_{2-x}Pb_xSr_2Ca_2Cu_2O_y$ is a promising candidate for superconducting applications operating around 100 K. Future work should explore microstructural characterisation at the nanoscale and test performance in real device configurations.

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